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FURTHER CHARACTERIZATION AND DEMULSIFICATION STUDIES ON OIL-IN-WATER EMULSIONS

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May 1986

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72 pp

ENERGY RESEARCH PROGRAM
COAL RESEARCH LABORATORY
DIVISION REPORT ERP/CRL 86-69(TR)

MICROMEDIA

FURTHER CHARACTERIZATION AND DEMULSIFICATION STUDIES ON OIL-IN-WATER EMULSIONS

BY

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ABSTRACT

The results of the preliminary screening and testing of ten new potential demulsifying chemicals (BC-51 to 60) supplied by Bayer AG Leverkusen at concentrations of 50,100,200 and 400 ppm are described. BC-56,57 and 58 showed good oil and water layer separation at concentrations of 200 and 400 ppm while BC-55 and 59 showed separation at 400 ppm when tested on a 5% oil-in-water emulsion. BC-56,57 and 58 were compared to five of the best demulsifiers (BC-17,19,21,22 and 47) of fifty previously tested Bayer chemicals by testing on oil-in-water emulsions containing different amounts of oil. At 5% oil-in-water, BC-19,21,22 and 47 showed good oil and water separation at 200 and 400 ppm like that observed for BC-56,57 and 58. BC-17 showed separation at 100 and 200 ppm with only poor separation at 400 ppm. At 7% oil-in -water, all the chemicals tested showed separation only at 400 ppm. At 5% oil-in-water, with a greater proportion of the oil contained in >38μm droplets, all these chemicals showed either good separation (BC-19,22,47,56,57 and 58) or separation starting (BC-21,58) at 100 ppm and continuing to 400 ppm. BC-17 showed good separation at 100 and 200 ppm with poor separation at 400 ppm.

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ÉTUDES COMPLÉMENTAIRES RELATIVES À LA CARACTÉRISATION ET À LA DÉMULSIFICATION DES ÉMULSIONS "HUILE DANS L'EAU"

par

S.P. Twa*

RÉSUMÉ

L'auteur décrit les résultats des essais et du classement préliminaires de dix nouveaux produits chimiques ayant un potentiel de démulsification (BC-51 à 60) présentés par Bayer AG Leverkusen à des concentrations de 50, 100, 200 et 400 ppm. Les produits chimiques BC-56, 57 et 58 ont démontré une séparation adéquate de la couche d'eau et d'huile à des concentrations de 200 et 400 ppm tandis que les produits BC-55 et 59 ont indiqué une séparation à 400 ppm quand ils ont été soumis à un essai dans une émulsion comprenant 5 % d'huile dans l'eau. Les produits chimiques BC-56, 57 et 58 ont été comparés à cinq des meilleurs agents démulsionnants (BC-17, 19, 21, 22 et 47) choisis parmi cinquante produits chimiques Bayer vérifiés antérieurement par des essais dans des émulsions "huile dans l'eau" contenant des quantités d'huile différentes . À un taux de 5 % d'huile dans l'eau, les produits BC-19, 21, 22, et 47 ont démontré une séparation adéquate de l'huile et de l'eau à 200 et 400 ppm, semblable à celle qui s'est produite avec les produits BC-56, 57 et 58. Le produit BC-17 a indiqué une séparation à 100 et 200 ppm et une séparation qui laissait à désirer à 400 ppm. Dans une émulsion contenant 7 % d'huile dans l'eau, tous les produits chimiques vérifiés ont indiqué une séparation seulement à 400 ppm. Dans une émulsion contenant 5 % d'huile dans l'eau et où une quantité plus importante d'huile se retrouvait dans les gouttelettes de >38 µm, tous ces produits chimiques ont dénoté une séparation adéquate (BC-19, 22, 47, 56, 57 et 58) ou un début de séparation (BC-21, 58) à 100 ppm se poursuivant jusqu'à 400 ppm. Le produit BC-17 a démontré une séparation adéquate à 100 et 200 ppm et une séparation minime à 400 ppm.

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INTRODUCTION

Bayer is involved in the manufacture of potential demulsifier chemicals. Work at the Coal Research Laboratory, Edmonton, has involved characterization and demulsification studies of chemicals supplied by Bayer on oil-in-water emulsions. Previous to this report, fifty chemicals had been examined with some showing very promising results. The work in this report describes the testing of ten additional Bayer Chemicals (OBW26451 - OBW26460). These ten chemicals, referred to as BC-51 to 60, were received on two different dates, the first seven on December 13, 1982 and the last three on July 5, 1983. The initial testing involved observing the effectiveness of the chemicals in separating oil-in-water emulsions into separate oil (or bitumen) and water layers. Further work sought to determine the minimum concentration at which some of the chemicals were effective on the emulsions used and to rate the effectiveness of the new chemicals with respect to one another as well as to the previously tested chemicals. As mentioned in the previous report, the criteria for rating the effectiveness of the chemicals is based on: a) whether the chemical shows a separation of oil and water layers, b) the dosage of the chemical that causes a separation, c) separation sharpness, i.e. the amount of water in the separated oil layer and the amount of oil left in the water layer, d) the time required for the separation and e) the quality of the separated oil layer (e.g. flocculated or creamed).

This report also describes work involved in testing the best demulsifiers of these sixty Bayer chemicals on oil-in-water emulsions containing different amounts of oil. This is intended not only to allow comparison studies between the demulsifiers using the same emulsions but also to show the actual effect of changes in emulsion oil content on the dosages required to cause oil and water separation.

EXPERIMENTAL

A. Emulsion

The emulsion used to evaluate BC-51 to 57 was an in-situ produced oil-in-water emulsion from the Fort McMurray, Alberta area and was composed of a number of individual samples produced from different wells.

The emulsion used was found to have a total bitumen content of 5.48% by weight as determined by gravimetric analysis (see Total Oil Analysis in the Analytical Procedures). The emulsion was tested with the Coulter Counter particle size analyzer (see the Analytical Procedures) and was found to contain 4.94% particulates (bitumen) with a MESD (Median Equivalent Spherical Diameter) of 9.61 μm (see Fig. 13). The discrepancy between the total bitumen content of 5.48% and the Coulter Counter value of 4.94% is mostly due to the prescreening of particulates coarser than 38μm that is necessary before running the emulsion sample on the Coulter Counter. The prescreening removes particulates larger than 38 μm which upon weighing were found to constitute 0.41% of the weight of the emulsion. Adding this value to that obtained from the Coulter Counter gives a total bitumen value of 5.35%, which is close to the value obtained by the total oil analysis (see Table 1). The water content of the emulsion was measured using a Mitsubishi Moisture Meter (see Analytical Procedures) and was found to be 93.26%. The % bitumen and % water in the emulsion were checked periodically over the period of testing and the results are reported in Table 2a.

Since BC58-60 were received at CRL at a later date, this necessitated preparing another emulsion in order to perform the testing. Another emulsion was prepared from a similar source and with a similar oil content (4.95%) to that used for testing BC-51 to 57 (see Table 2b and Fig. 31). It should be emphasized though that there are still differences between the two emulsions (e.g. compare MESDs in Tables 2a and 2b) and these will have some effect on the demulsifiers' effectiveness. However, these effects are not thought to be significant enough to affect the ability to rate the demulsifiers as generally "good" or "bad" with respect to one another.

This second emulsion was also used in the testing of the best of the Bayer demulsifiers (see the section on Further Demulsifier Comparison

Table 1 - % Oil and Water Analysis of Stock Emulsion used to test BC-51 to 57

Method	% Bitumen	% Water
Coulter Counter (<38 μm)	4.94 ┐ 5.35	*94.65
Oversize (>38 μm)	0.41 ┘	
Gravimetric Analysis	5.48	*94.52
Moisture Meter	*6.74	93.26

*Calculated by difference from 100%

- See Figure 13 for the initial size distribution plot obtained from the Coulter Counter analysis of the stock emulsion.

Table 2 - Analyses of Stock Emulsions with Time

a) Emulsion used to evaluate BC-51 to 57

DATE (1983)	% BITUMEN (by CC <38 μm)	% BITUMEN (>38 μm)	% BITUMEN (by GA)	MESD (from CC, μm)	% H_2O (by MM)
Jan 25,26	4.94	0.41	5.48	9.61	93.26
Feb 4,9	4.60	-	5.54	9.23	94.48
Feb 14,16	6.20	-	6.12	8.80	95.55
Feb 21	6.34	0.34	7.24	8.70	92.68
Feb 28	7.15	0.91	8.40	8.86	-

b) Emulsion used to evaluate BC-58 to 60

DATE (1983)	% BITUMEN (by CC <38μm)	% BITUMEN (>38 μm)	% BITUMEN (by GA)	MESD (from CC, μm)	% H_2O (by MM)
June 20	4.95	-	5.1	7.44	94.25
June 27	4.94	-	5.6	7.39	-
July 6	4.77	-	-	7.08	-

CC = Coulter Counter
GA = Gravimetric Analysis
MM = Moisture Meter

Studies) and was also one of the emulsions used to examine the effect of different oil contents on demulsifier performance.

B. Demulsifier

The demulsifier solutions were prepared by diluting the received chemicals in deionized water to give solutions of 0.3% active chemical by weight. Water was weighed into a 100 ml beaker and then the required amount of demulsifier was added to make up the solution to a total of 100g (see Table 3). The solutions were stirred until complete dissolution was reached as judged by solution homogeneity. The chemical solutions to be used were freshly prepared each day. BC-60 was received as an off-white, translucent, semi-solid and did not totally dissolve in water, even after stirring overnight.

C. Test Procedure

The demulsifier testing was done in 50 ml capped glass culture tubes. Thirty ml of emulsion were pipetted into each tube. Since the prepared demulsifier solutions contained 0.3% active chemical, addition of 1 ml of demulsifier solution would correspond to 100 ppm with respect to the 30 ml of emulsion. The tests involved observing the effect of up to 400 ppm of demulsifier so up to 4 ml of demulsifier solution was required. To balance the total volume of liquid in tubes containing less than 400 ppm demulsifier, deionized water was added such that the total volume of demulsifier solution and deionized water equalled 4 ml (see Table 4). Thus, after adding the emulsion to the culture tubes, the required amount of deionized water was added. This was followed by the addition of 3 ml toluene to each culture tube which assists in solubilizing the bitumen in the emulsion. This volume and choice of solvent was determined with respect to the results obtained in the previous Bayer report. Finally, after the solvent addition, the determined amount of demulsifier solution was added to give the desired dosage.

After all the reagents were added, the culture tubes were capped, placed in a support rack and inverted ten times to mix the contents. The tubes were then quickly placed in a circulating water bath preheated to 60°C and a timer was started. Photographs were taken of the tubes at specific time intervals starting at 15 seconds and ending at 24 hours.

Table 3 - List of Chemicals Tested and Amounts Required of Each to Prepare Solutions Containing 0.3% Active Chemicals.

EMR No.	BAYER No.	ACTIVE CHEMICALS(%)	CHEMICAL ADDED (g)	WATER (g)
BC-51	OBW 2642/51	17	1.76	98.24
BC-52	OBW 2642/52	14.2	2.11	97.89
BC-53	OBW 2642/53	16.7	1.80	98.20
BC-54	OBW 2642/54	16.5	1.82	98.18
BC-55	OBW 2642/55	24.3	1.23	98.77
BC-56	OBW 2642/56	24.9	1.20	98.80
BC-57	OBW 2642/57	22.5	1.33	98.67
BC-58	OBW 2642/58	28.1	1.07	98.93
BC-59	OBW 2642/59	30.7	0.98	99.02
BC-60	OBW 2642/60	33.0	0.91	99.09

Table 4 - Volume of Reagents Used in Initial Screening Test

CULTURE TUBE No.	DEMULSIFIER DOSAGE(ppm)	EMULSION (ml)	TOLUENE (ml)	H_2O (ml)	REAGENT SOLUTION (ml)
1	0	30	3.0	-	-
2	0	30	3.0	4.0	-
3	50	30	3.0	3.5	0.5
4	100	30	3.0	3.0	1.0
5	200	30	3.0	2.0	2.0
6	400	30	3.0	-	4.0

This procedure was changed slightly for the testing of BC-58 to 60 and subsequent tests. In this case the timer was started just before the chemicals were mixed. Since the mixing requires about 15 seconds, times for BC-51 to 57 are actually 15 seconds longer than the labelled time if the demulsification reaction is deemed to start as soon as mixing begins. This was the reason for changing the timing procedure.

D. Initial Screening

In order to be able to compare results, the ten new chemicals (BC-51 to BC-60) were tested by the same procedures as those followed for the other 50 Bayer chemicals previously tested (BC-1 to BC-50). Exact duplication of test conditions was not possible because new stock emulsions were prepared and used for this testing. The bitumen content of these emulsions was different than that for the emulsions used previously.

Initial screening of the chemicals involved testing the effectiveness of each chemical in separating oil and water in an emulsion at four different concentrations. The concentrations tested were 50, 100, 200 and 400 ppm, based on the total volume of emulsion used. Two blanks were included with each test for comparison purposes. The first blank contained emulsion only, while the second contained emulsion, deionized water and toluene. In total, six culture tubes were prepared for each test and after being mixed, were photographed over a period of 24 hours.

E. Concentration Optimization

The aim of the initial screening is to identify any good demulsifiers out of the 10 new chemicals. This was followed by an optimization of the concentration by more systematically varying the chemical dosage in the defined concentration range (e.g. by 10 ppm increments) for selected chemicals.

F. Further Demulsifier Comparison Studies

The characteristics of the stock emulsion changes over time. This makes accurate comparison difficult. In order to gain a better idea on the effect of such changes on demulsifier performance, three stock emulsions, containing approximately 3, 5 and 7% oil with droplet sizes smaller than 38 µm were prepared. The demulsifiers tested on these stock emulsions would be

some of those that showed the best demulsification of all the sixty Bayer chemicals received. These would include BC-17, 19, 21, 22, and 47, in addition to the best of BC-51 to 60.

RESULTS AND DISCUSSION

A. Initial Screening

In order to compare the results for BC-51 to 57 to previous results (for BC-1 to 50), BC-47 was retested on the emulsion used to test BC-51 to 57. In the previous report, when an emulsion with about 6% bitumen content was used, BC-47 showed good oil and water separation starting at a 100 ppm dosage during the initial screening test. Separation was first noted within 15 seconds of the start of the test. More accurate testing a few weeks later however, showed separation first appearing between 110 and 120 ppm. The reason for this difference was thought to be due in part to the gradually changing characteristics of the emulsion (i.e. increasing % bitumen). In this report, when BC-47 was initially tested on the emulsion used for testing BC-50 to 57, which contained about 5.5% bitumen, BC-47 didn't show separation until 200 ppm (see Fig. 8), with separation first noted within 30 seconds. This testing though, had commenced several weeks after the emulsion had been prepared. The results continue to suggest that as would be expected, the % bitumen making up the emulsion is a factor in determining not only effective demulsifier dosage, but also the speed of oil and water layer separation. BC-47 was also used as a control during the testing of BC-58 to 60 (see Fig. 25) which was carried out using the same emulsion. Again, separation was only observed at 200 at 400 ppm BC-47.

Of the ten chemicals tested (BC-51 to BC-60), only five showed oil and water separation after 24 hours (see Table 5 and Figures 1-7 and 28-30). The emulsion was effectively broken at 400 ppm for BC-55 and 59 and at 200 and 400 ppm for BC-56, 57 and 58. BC-55 and 59 showed layer separation within 2 minutes of starting while BC-56, 57 and 58 showed separation within one minute. Table 6 shows the results of Coulter Counter and Moisture Meter analyses of samples that showed separation. The size distribution plots for the residual oil contained in the water layer for some of these samples can be seen in Figures 14 to 20, 32 and 33. From the above results BC-56 and

Table 5 - Relative Degree of Flocculation and Water Clarity for Separated layers

CHEMICAL	200 ppm	400 ppm
BC-55	-	⊕ XX
BC-56	⊕ XXX	⊕ X
BC-57	⊕ XXX	⊕ X
BC-47	⊕ XXX	◕ X
BC-58	⊕ XX	⊕ X
BC-59	-	⊕ XX
BC-47	◕ X	⊕ X

LEGEND

Clarity
⊕ = Clear
⊕ = Slight cloudiness
◕ = Cloudy
● = Milky or opaque

Flocculation
X = None or very little
XX = Some flocculation
XXX = More flocculation
XXXX = All flocculation

Table 6 - Analyses of Reagent Solutions Showing Good Separation

Sample	Water Layer - by CC		Oil Layer - by MM % H_2O *
	MESD (μm)	% Oil	
BC-55 400 ppm	7.46	0.0011	7.35
BC-56 200 ppm	29.53	0.0019	31.95
BC-56 400 ppm	15.55	0.0006	0.30
BC-57 200 ppm	20.44	0.0019	25.92
BC-57 400 ppm	12.25	0.0004	0.13
BC-47 200 ppm	13.59	0.0038	87.59
BC-47 400 ppm	9.48	0.0007	39.01
BC-56 120 ppm	15.15	0.5152	89.96
BC-56 130 ppm	19.88	0.1083	59.44
BC-56 140 ppm	20.76	0.0916	84.80
BC-56 150 ppm	20.60	0.0815	63.48
BC-57 110 ppm	15.49	2.368	85.24
BC-57 120 ppm	9.55	1.162	89.27
BC-57 130 ppm	11.98	0.0193	88.87
BC-57 140 ppm	19.77	0.0252	90.26
BC-57 150 ppm	20.66	0.0325	64.92
BC-47 160 ppm	6.52	0.0264	80.22
BC-47 170 ppm	5.53	0.0229	78.00
BC-47 180 ppm	6.33	0.0095	85.09
BC-47 190 ppm	12.39	0.0090	76.04
BC-47 200 ppm	7.31	0.0066	79.46
BC-47 210 ppm	24.40	0.0124	75.31
BC-47 220 ppm	11.16	0.0047	43.73
BC-47 230 ppm	23.10	0.0051	10.08
BC-47 240 ppm	7.83	0.0033	0.158
BC-47 250 ppm	21.38	0.0044	0.078
BC-58 200 ppm	-	-	29.16
BC-58 400 ppm	14.12	0.0020	0.21
BC-59 400 ppm	27.53	0.0023	5.18

CC = Coulter Counter

MM = Moisture Meter

*High percentages suggest high degree of flocculation

BC-57 were chosen from BC-51 to 57 for further testing to determine the minimum dosage required to cause layer separation.

B. Concentration Optimization

From the initial screening tests it was shown that for reagents BC-56 and BC-57 the lowest effective demulsifier dosage was 200 ppm. Since no separation was observed at 100 ppm the minimum required dosage for these chemicals is somewhere between 100 ppm and 200 ppm. In order to determine the minimum demulsifier dosage more accurately, experiments were run in which the concentration of demulsifier was varied more gradually. These tests examined concentrations between 60 and 150 ppm, which varied by 10 ppm increments. These tests were similar to the initial screening tests, the only difference being that 10 tubes and no blanks were used rather than 6 tubes with blanks.

The 24 hour photographs from the optimization tests showed an oil and water separation starting at about 100 ppm for both BC-56 and BC-57 (see Figures 9 and 10) with good separation noticeable at 120ppm. Table 6 shows the results of Coulter Counter and Moisture Meter analyses of samples that showed good separation.

In the dosage optimization tests BC-56 and BC-57 gave very similar results. Both chemicals seem to have a similar effectiveness in oil and water separation. In order to compare the effectiveness of these two chemicals to the chemicals tested in previous reports (BC-1 to BC-50), an optimization test was also done on BC-47. A ten tube test was first done with the reagent concentration ranging from 60 ppm to 150 ppm. This test appeared to show separation becoming effective only around 150 ppm (see Figure 11). A second test was done with dosages of 160 ppm to 250 ppm. The results from this test showed that the minimum concentration of BC-47 needed to cause an oil and water separation was 160 ppm (see Figure 12). The previous report showed good separation between 100 and 120 ppm for BC-47. As mentioned earlier, the stock emulsion does change over time. This may be due to the fact that a thick sludge deposit slowly forms on the bottom of the emulsion container and the amount of this deposit increases the longer the emulsion is left in its container. Stirring does not put all of this deposit back into solution so that it tends to accumulate. As the upper layer of the emulsion is used up in the initial screening tests the emulsion

left appears to increase in bitumen content as noted in Table 2a. The concentration optimization tests are done after the initial screening tests and it is usually then that differences in the dosage required for oil and water separation are observed. Thus, using emulsions with different % bitumen does appear to affect the effectiveness of the chemical demulsifiers tested. Since BC-47 had been observed to separate at 100 ppm, the range observed for the start of separation for BC-47 is about 50 ppm since the concentration optimization done in this report showed separation starting at 150 ppm. If this variability is due to the % bitumen in the emulsion, this is a fairly significant effect on demulsifier dosage. Nevertheless, as compared to many of the chemical demulsifiers tested, many of which show no oil and water separation at a 400 ppm dosage, BC 47, 56 and 57 appear to be among the best demulsifiers tested (for the 60 chemicals received from Bayer). All that can be safely concluded at this point is that BC-47, 56 and 57 are fairly similar in effectiveness and that further tests are required to determine whether BC-56 and 57 are inferior to BC-47.

C. Further Demulsifier Comparison Studies

As a result of the findings of the initial screening, BC-56, 57 and 58 were included in the testing of BC-17, 19, 21, 22 and 47 on 3, 5 and 7% oil-in-water emulsions.

The 5% oil stock emulsion was the same emulsion as that used to test BC-58 to 60 (see Table 2b) and therefore the results of the tests on BC-58 to 60 can be compared to those for BC-17, 19, 21, 22, 47, 56 and 57. The characteristics of the 7 and 3% oil emulsions are as shown in Table 7a and 7b, respectively. Note that the 3% oil emulsion, while it did contain about 3% oil in droplets less than 38 μm in diameter, had a total oil content of between 5 and 6%. Thus, oil droplets larger than 38 μm were present. This differs from both the 5 and 7% emulsions in which almost all of the oil droplets in these emulsions were smaller than 38 μm in size.

The results of the test with the 5% emulsion are shown in Figures 21-30. As mentioned previously, BC-58 showed separation at 200 and 400 ppm. This was also true for BC-19, 21, 22, 47, 56 and 57. BC-17, as seen in earlier reports, showed separation at 100 and 200 ppm with poor to no separation at 400 ppm. Table 8 shows the % oil found in the separated water layers. These % oil results give further information on the effectiveness

Table 7 - Analyses of Stock Emulsions used in Comparison Studies

a) 7% bitumen emulsion

DATE (1983)	% Bitumen (by CC, <38 μm)	% Bitumen (by GA)		MESD (from CC, μm)
		<38μm	TOTAL	
July 4, 5	7.39	6.63	6.87	8.09
July 7	7.23	7.55	8.19	8.26

b) 3% bitumen emulsion

DATE (1983)	% Bitumen (by CC, <38 μm)	% Bitumen (by GA)		MESD (from CC, μm)
		<38μm	TOTAL	
July 11, 12	3.21	3.01	5.35	8.98
July 13	-	2.90	6.30	-

CC = Coulter Counter
GA = Gravimetric Analysis

Table 8 - Analyses of Reagent Solutions Showing Good Separation from Comparison Studies

Sample	% Oil in Water Layer (by CC)		
	5% emulsion	7% emulsion	3% emulsion
BC-17 100 ppm	0.0088	-	0.0048
200 ppm	0.0007	-	0.0020
400 ppm	-	0.0018	-
BC-19 100 ppm	-	-	0.0068
200 ppm	0.0746	-	0.0028
400 ppm	0.0023	0.0134	0.0009
BC-21 100 ppm	-	-	0.0145
200 ppm	0.0094	-	0.0022
400 ppm	0.0076	0.4225	0.0037
BC-22 100 ppm	-	-	0.0063
200 ppm	0.0006	-	ND
400 ppm	0.0002	0.0248	0.0011
BC-47 100 ppm	-	-	0.0071
200 ppm	0.0004	-	0.0063
400 ppm	0.0002	0.0248	0.0011
BC-56 100 ppm	-	-	0.0084
200 ppm	0.0010	-	0.0063
400 ppm	0.0002	0.0115	0.0066
BC-57 100 ppm	-	-	0.0236
200 ppm	0.0008	-	0.0077
400 ppm	0.0002	0.0133	0.0011
BC-58 100 ppm	-	ND	-
200 ppm	ND	ND	0.0009
400 ppm	0.0020	ND	0.0021

CC = Coulter Counter

ND = Not done

- = no clear separation noted

of separation. For example, the separation in culture tube 5 (200 ppm) for BC-19 (fig. 22) looks a little poor. This is confirmed in Table 8 which shows a relatively high oil content in the water layer as compared with the results at 200 ppm for the other demulsifiers. There is a high variability in these % oil results (compare the two different values obtained for 200 ppm BC-47 in Table 6) but they are useful in adding to the information available from the photographs and in giving a general relationship between demulsifiers and their different concentrations. Therefore, the results show a general similarity between 7 of the 8 demulsifiers tested with BC-17 being the only one showing really different behavior.

The results obtained using the 7% oil emulsion can be seen in Figures 34-40. These tests kept to the same test procedure that was used for the 5% emulsion testing. The amount of toluene added was kept at an approximate 2:1 toluene to total oil ratio. For example, 4.5 ml of toluene was added to culture tubes containing about 2.1 ml (7% of 30 ml) of oil or bitumen.

Much poorer separation was observed when this higher oil containing emulsion was used. BC-17 showed good separation but only at 400 ppm. BC-19, 22, 47, 56 and 57 also showed separation at 400 ppm but as the photographs and Table 8 indicate, the separated water layers are much darker and contain higher oil contents than that observed when the 5% bitumen emulsion was used. Though it is difficult to see, BC-21 does show some separation at 400 ppm though the water layer is still quite opaque. BC-58 was not tested on this 7% emulsion.

Tests on the demulsifiers using the 3% oil emulsion showed better separation than that using the 5% emulsion (see Figs. 41-48). In fact, most of the reagents tested showed good, clear separation at 100, 200 and 400 ppm dosages. Exceptions were BC-17, showing no separation at 400 ppm and BC-21 and 58, both of which showed only poor separation 100 ppm. This test emulsion contains about the same percent total oil (5-6%) as that found in the 5% emulsion (see Table 2b). In this case though, the former contained only about 3% of the oil in droplets smaller than 38 μm in diameter while the latter contained about 5% of the oil in this size range. The amounts of toluene used were the same in both cases (i.e. 3 ml per culture tube). The demulsifiers, however, definitely worked at a much lower concentration (i.e. at 100 ppm vs 200 ppm) when the 3% emulsion was used. The two emulsions

used were from similar, though different sources, and while this could give rise to several explanations as to why the demulsifiers worked better on the 3% emulsion, one of the most reasonable explanations concerns surface area to volume ratios. Since demulsifiers are surface-active agents, a lower surface area should require less demulsifier. Thus, effective oil and water layer separation should occur at a lower demulsifier concentration for the emulsion having larger droplets, other factors being similar.

Thus, from the comparisons made above, it has been shown that with the exception of BC-17, the demulsifiers BC-19, 21, 22, 47, 56, 57 and 58 are quite similar in effectiveness on the emulsions tested. BC-17 is the most effective reagent at the lowest concentration and tends to break oil-in-water emulsions before any of the other demulsifiers. However, the effectiveness of BC-17 appears to be over a narrower range than the range observed for the other chemicals such that at higher concentrations BC-17 does not do as good a job of separating oil and water layers.

The sixty Bayer chemicals tested were received in four groups; BC-1 to 43, BC-44 to 50, BC-51 to 57 and BC-58 to 60. These were received September 1, 1981, August 25, 1982, December 13, 1982 and July 5, 1983. As with many reagents, degradation can be expected to occur with time and therefore, it is reasonable to expect that at least the earlier demulsifiers have changed somewhat in composition from when they were first tested. How much this change has altered the demulsifiers' efficiency is impossible to judge, but the above results show that the chemicals tested are still quite effective and their range of effectiveness is very similar to that found when they were first tested.

The results obtained during the demulsifier testing were used to judge general relationships between the chemicals and give some idea on their effectiveness over the ranges tested. Further testing should involve looking at higher dosages and though these would not be economical on an industrial scale, they may give more information on the range of usefulness of the chemicals. Ideally, a demulsifier should break the emulsion at higher concentrations if for example, dosage is being varied to try and compensate for the varying oil content in the emulsion. As seen with BC-17, a 400 ppm dosage works on a 7% oil containing emulsion but does not work for a 5% oil containing emulsion. At first glance these results seem strange. If this was being observed in an industrial treatment plant, unless the

useful range of the demulsifier is known and carefully controlled, a practice of "If it does not work...add more" will be an expensive waste of time. Therefore, to minimize problems such as these, demulsifiers having a good working range are desirable so that overdosing is not so harmful to the separation process.

CONCLUSIONS

Five of the ten new Bayer chemicals received (BC-51 to 60) showed oil and water layer separation over the range of dosages tested on a 5% oil-in-water emulsion. BC-56, 57 and 58 showed separation from between 100 and 200 ppm to at least 400 ppm while BC-55 and 59 showed separation only from between 200 and 400 ppm to 400 ppm.

A group of the most promising demulsifiers, which included BC-17, 19, 21, 22, 47, 56, 57 and 58 were tested on oil-in-water emulsions containing different amounts of oil. The greater the oil content, the higher the concentration of demulsifier required to cause oil and water layer separation. These demulsifiers were also found to be more effective on emulsions containing a higher percentage of oil droplets larger than 38 µm. All these chemicals were similar in effectiveness except for BC-17 which was found to be the most effective reagent at the lowest concentration even though it possibly had the narrowest range of effectiveness since it showed poor layer separation a higher dosages.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the work of S.Bresciani (University of Regina) and M. Brule (University of Victoria) who assisted with the work on the emulsion and demulsifier testing and analyses during their co-op work terms (January - April and May - August, 1983, respectively).

FIGURES

#1 to #48

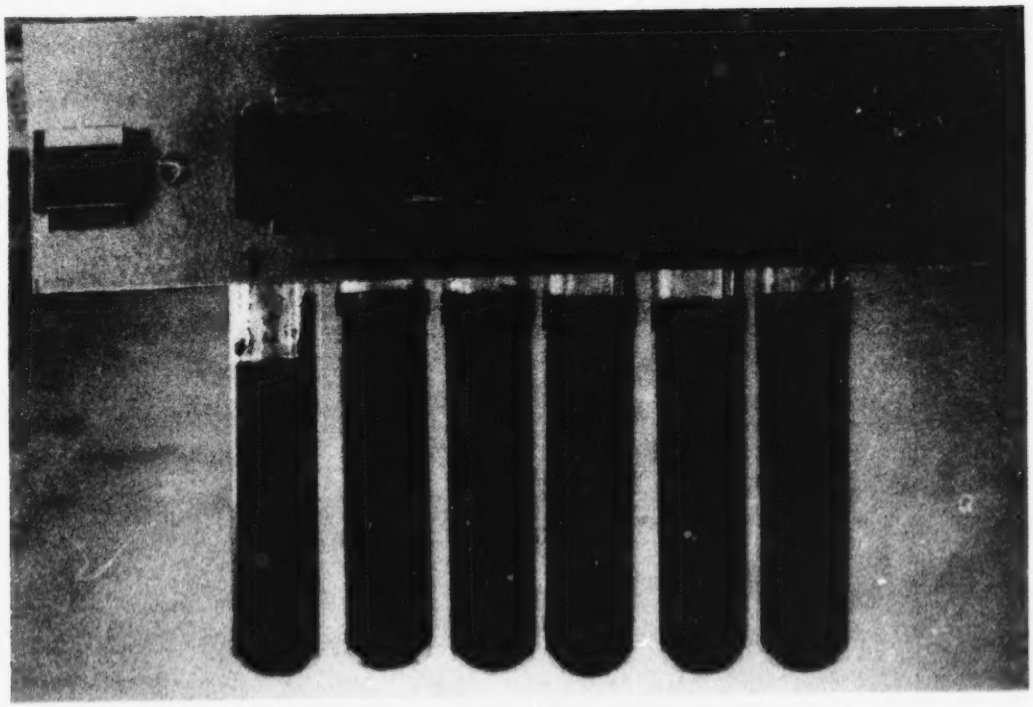


Figure 1: Initial Screening BC-51

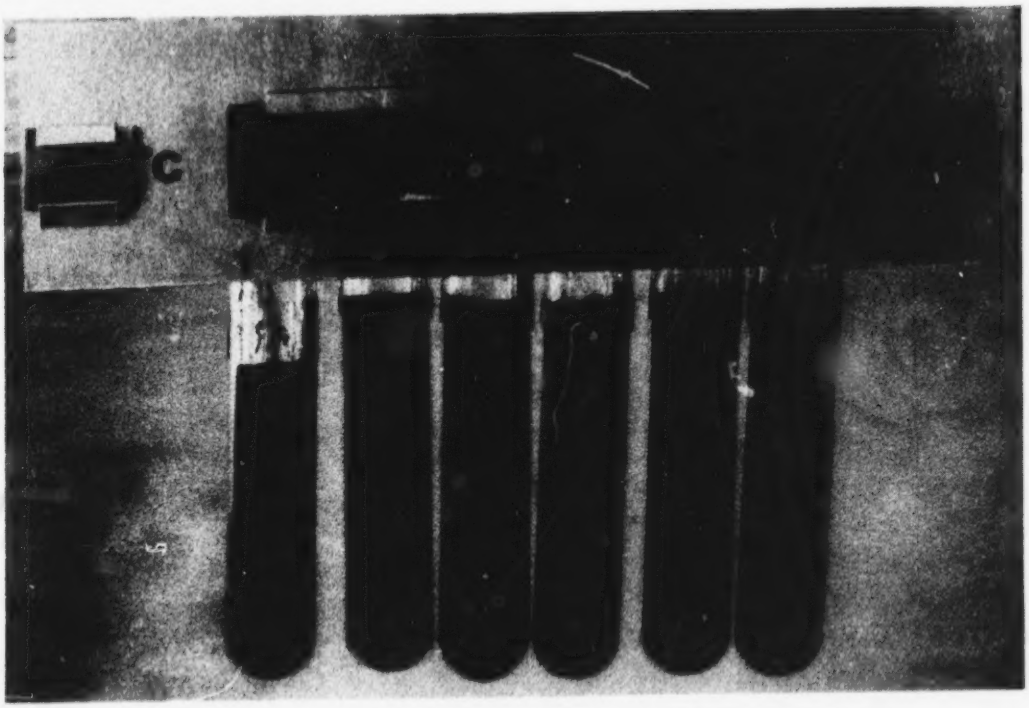


Figure 2: Initial Screening BC-52

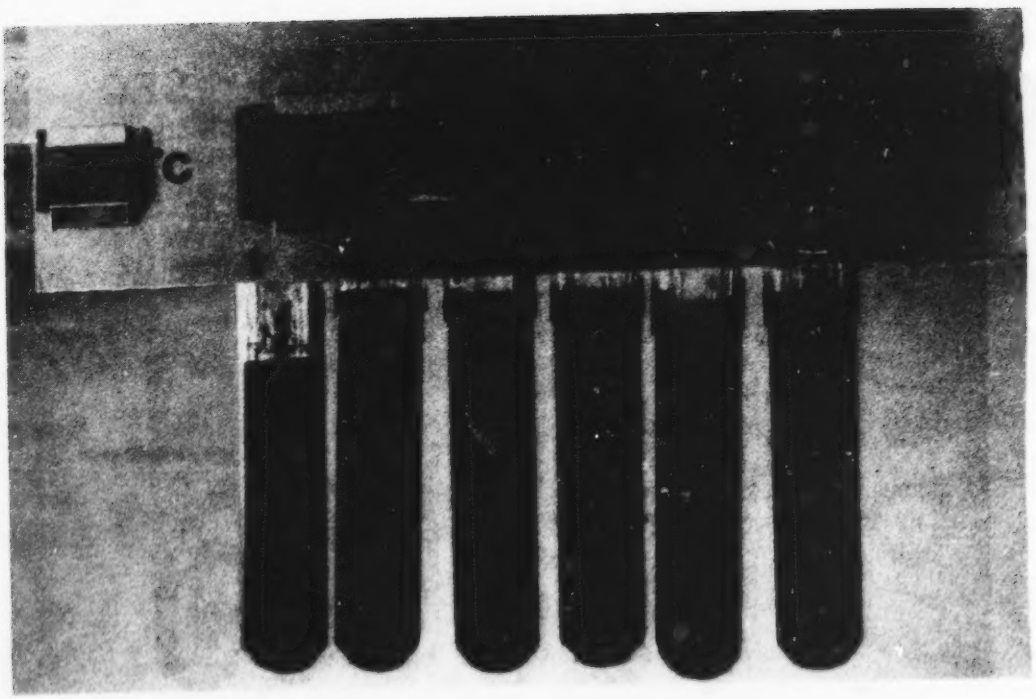


Figure 3: Initial Screening BC-53

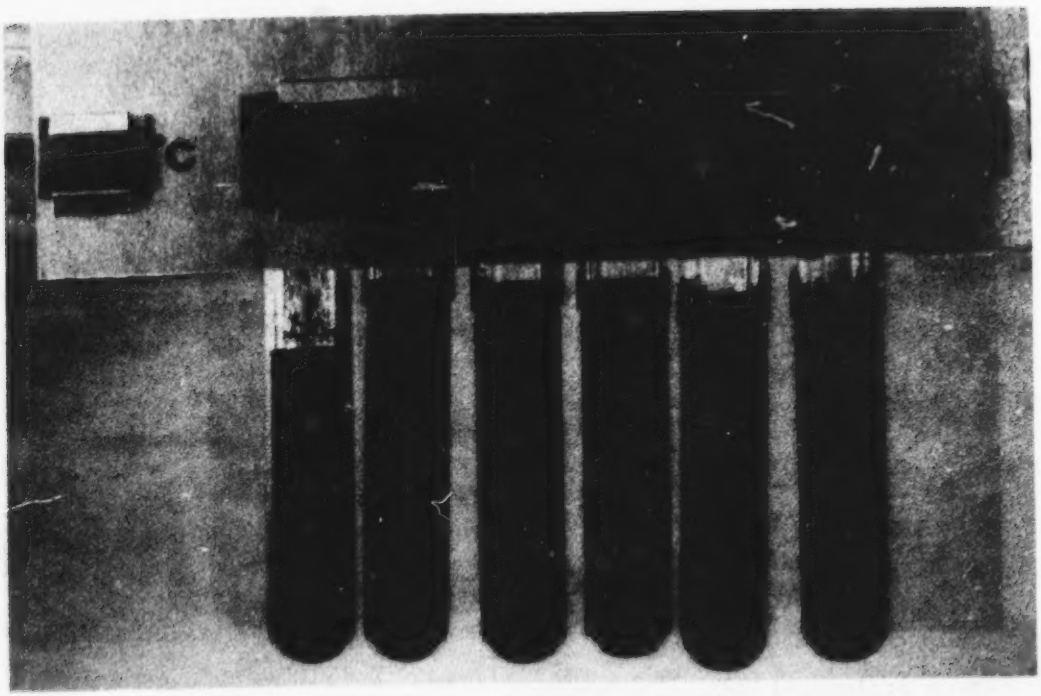


Figure 4: Initial Screening BC-54

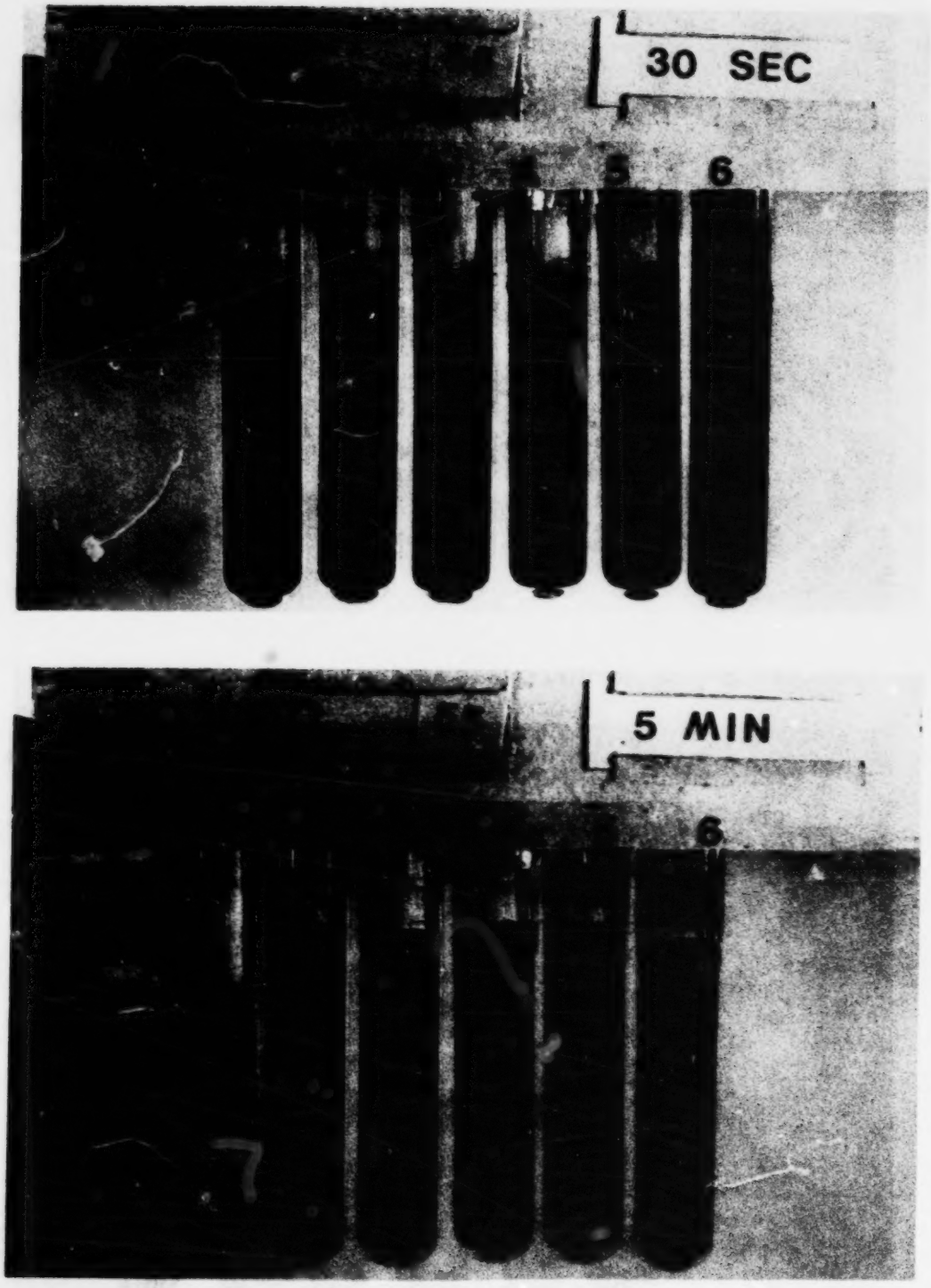
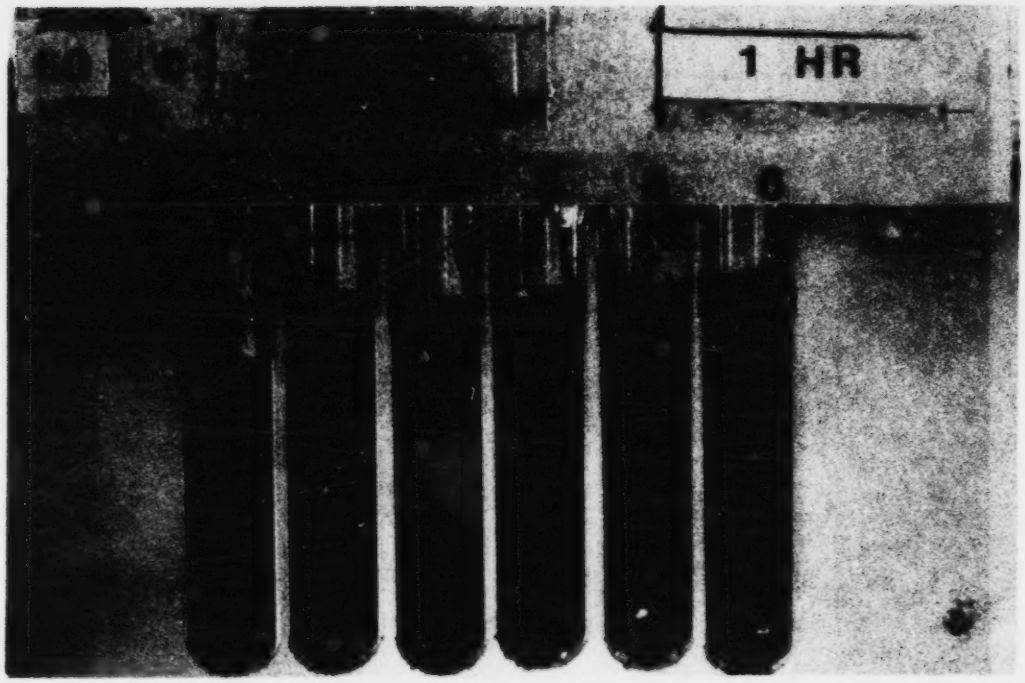


Figure 5: Initial Screening BC-55


1 HR

24 HR

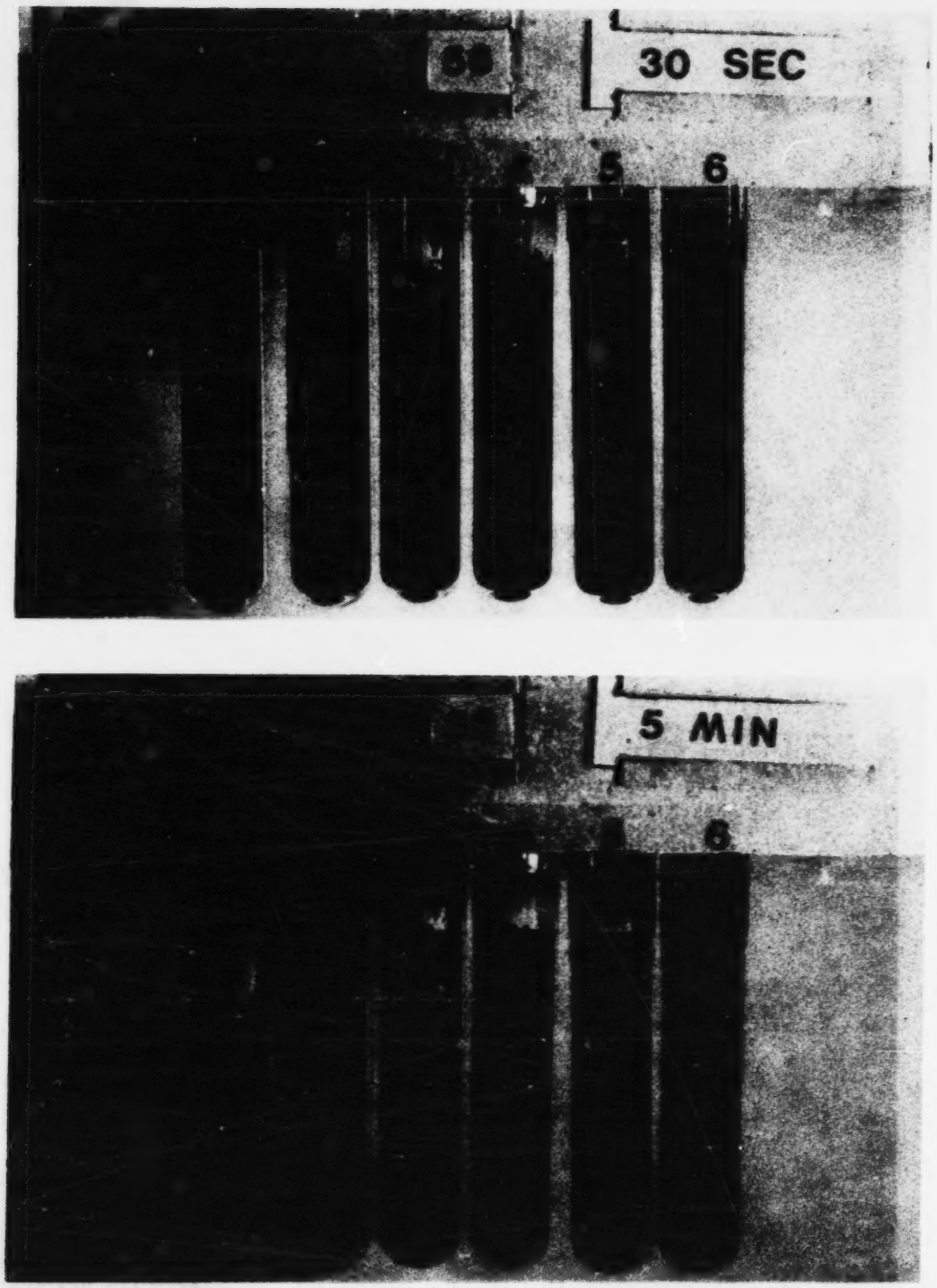
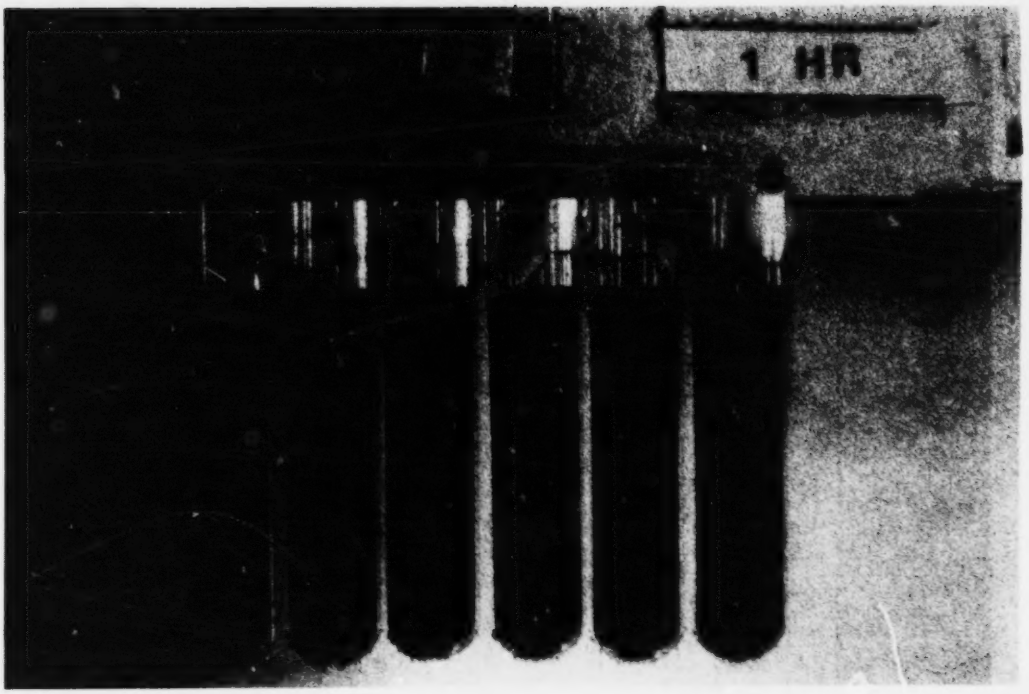
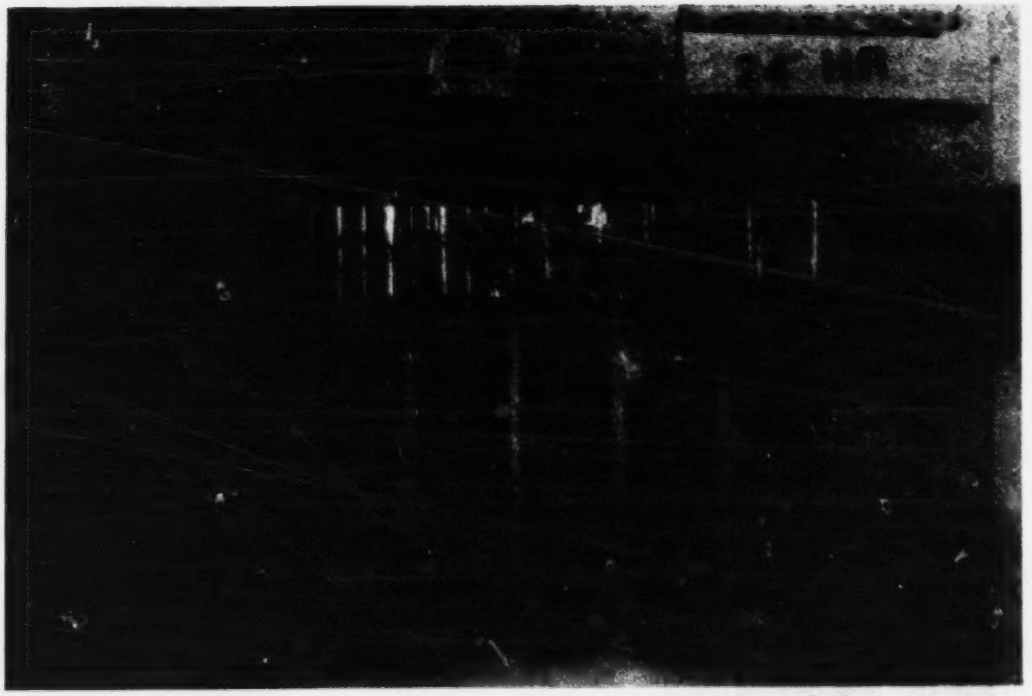


Figure 6: Initial Screening BC-56


1 HR



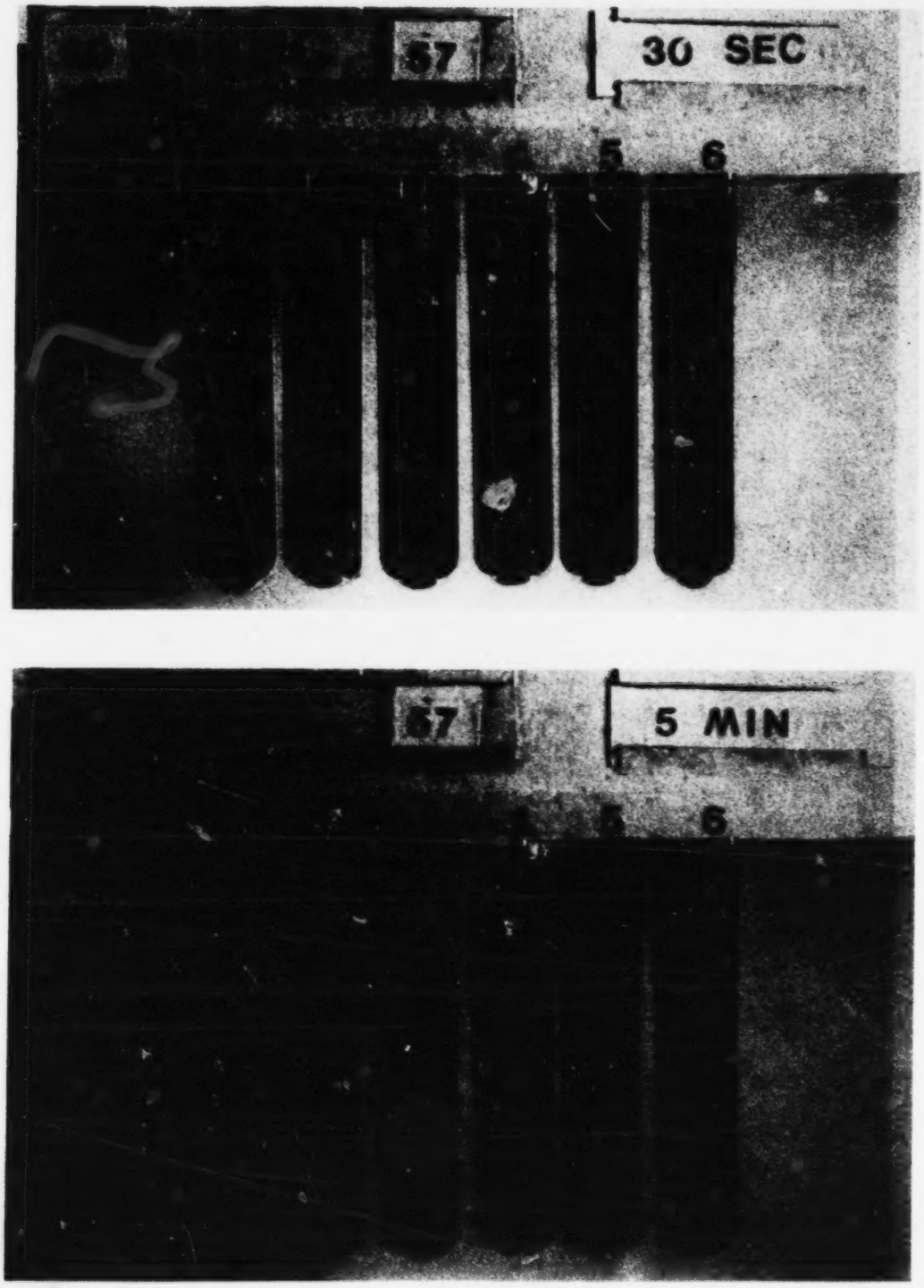
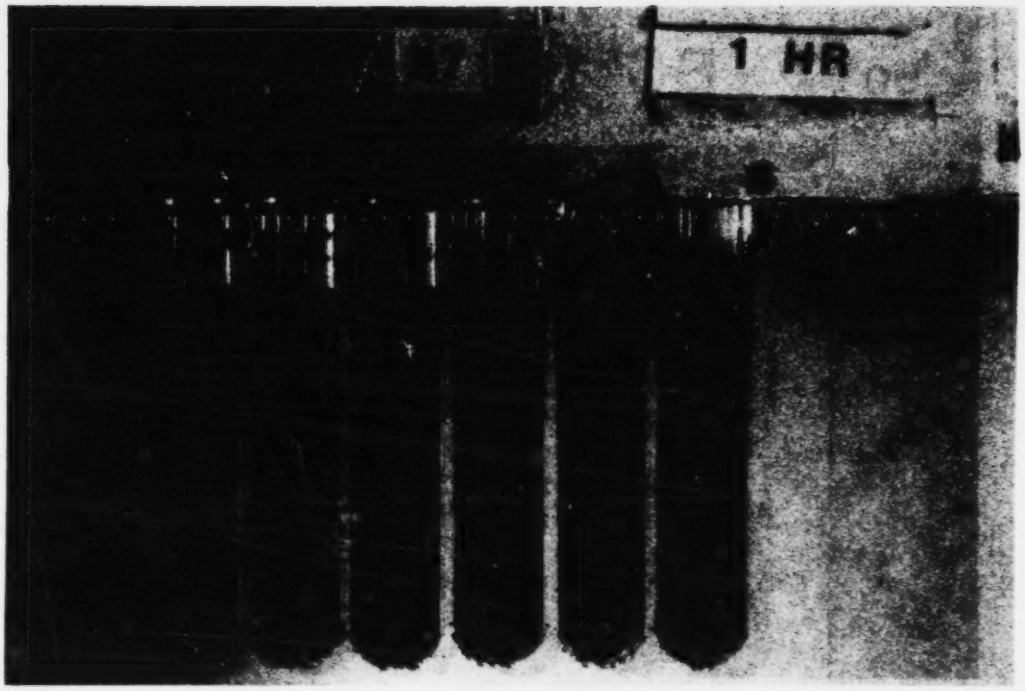
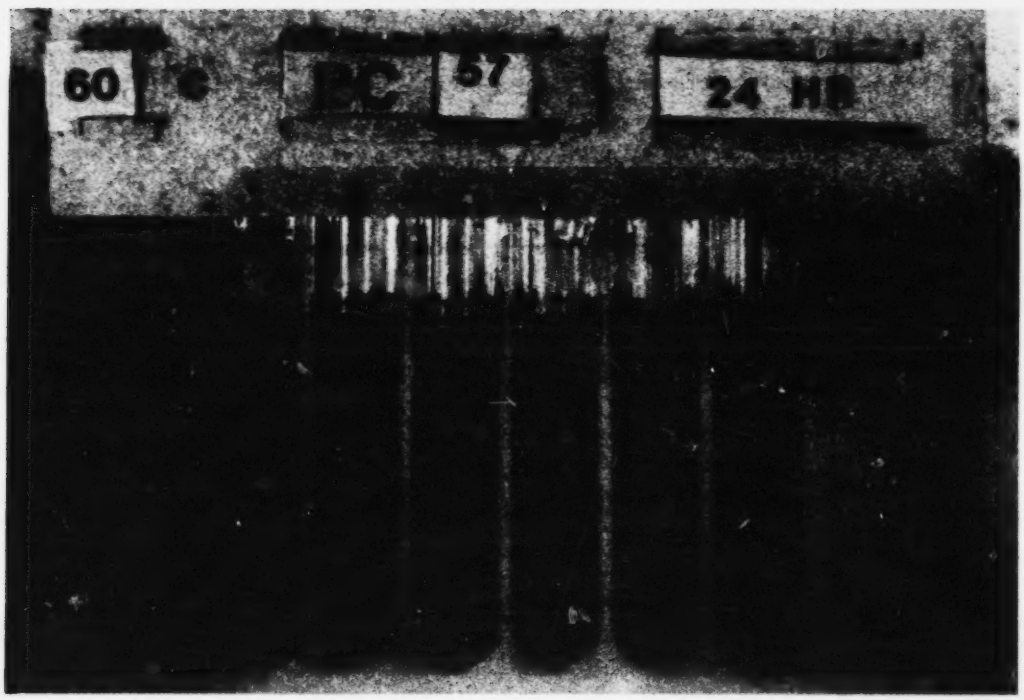


Figure 7: Initial Screening BC-57


1 HR


60
57
24 HR

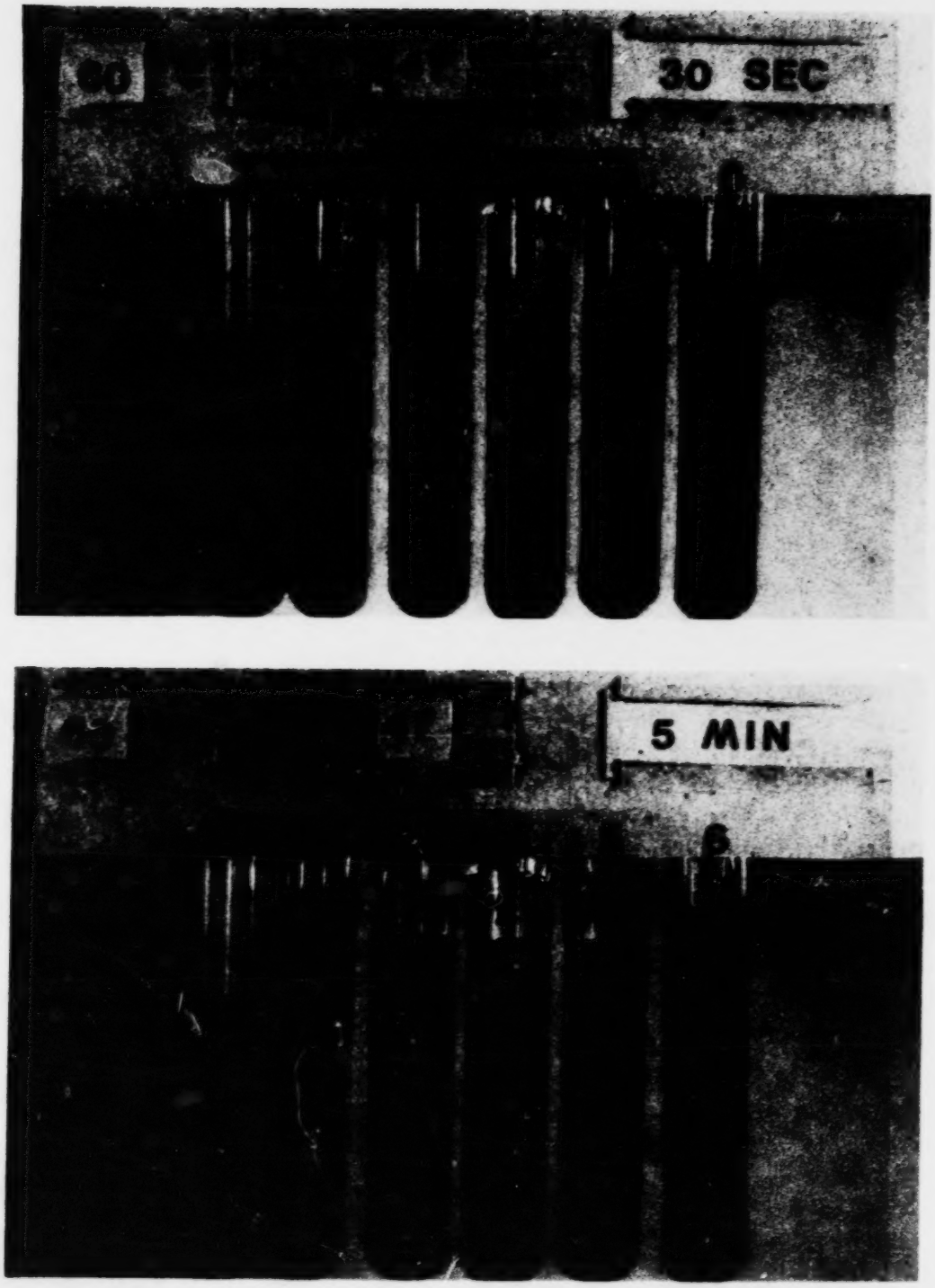
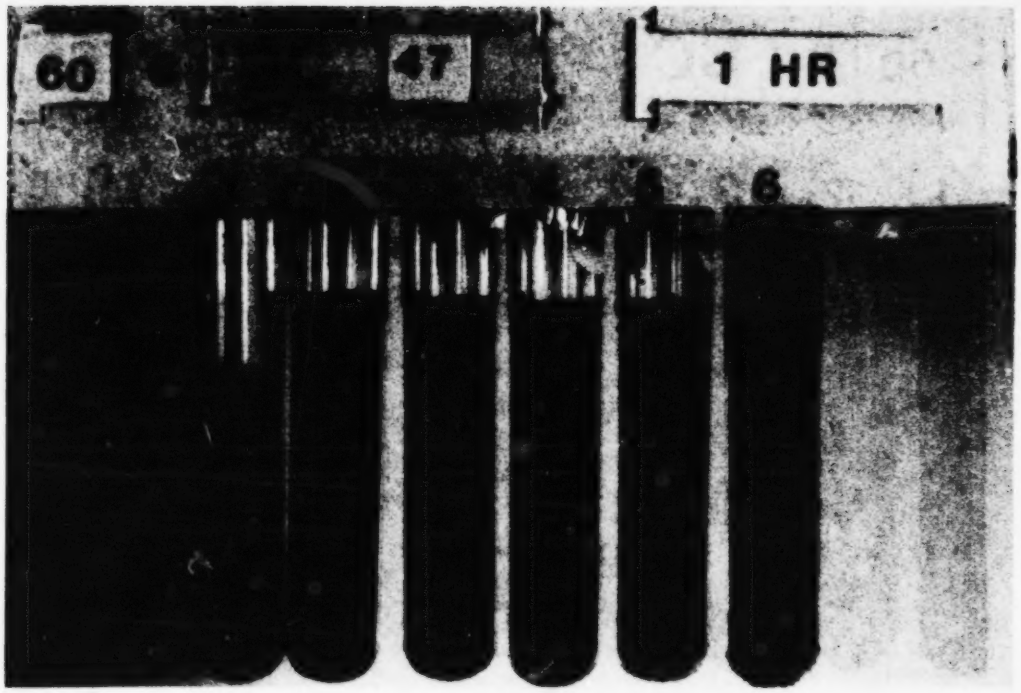
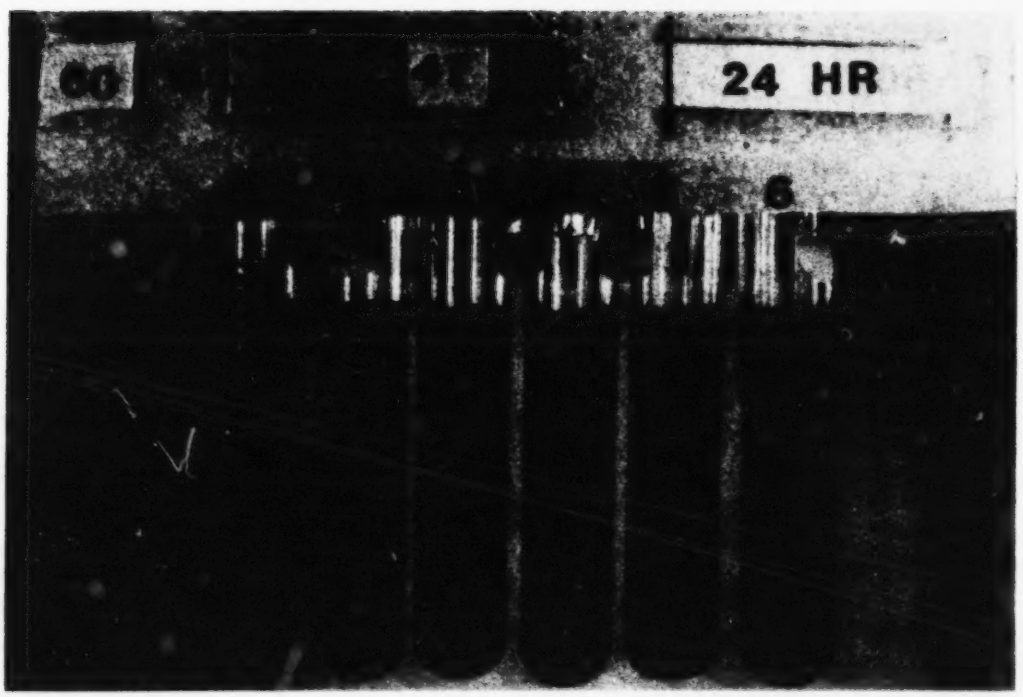


Figure 8: Initial Screening BC-47


60
47
1 HR
6


24 HR
6

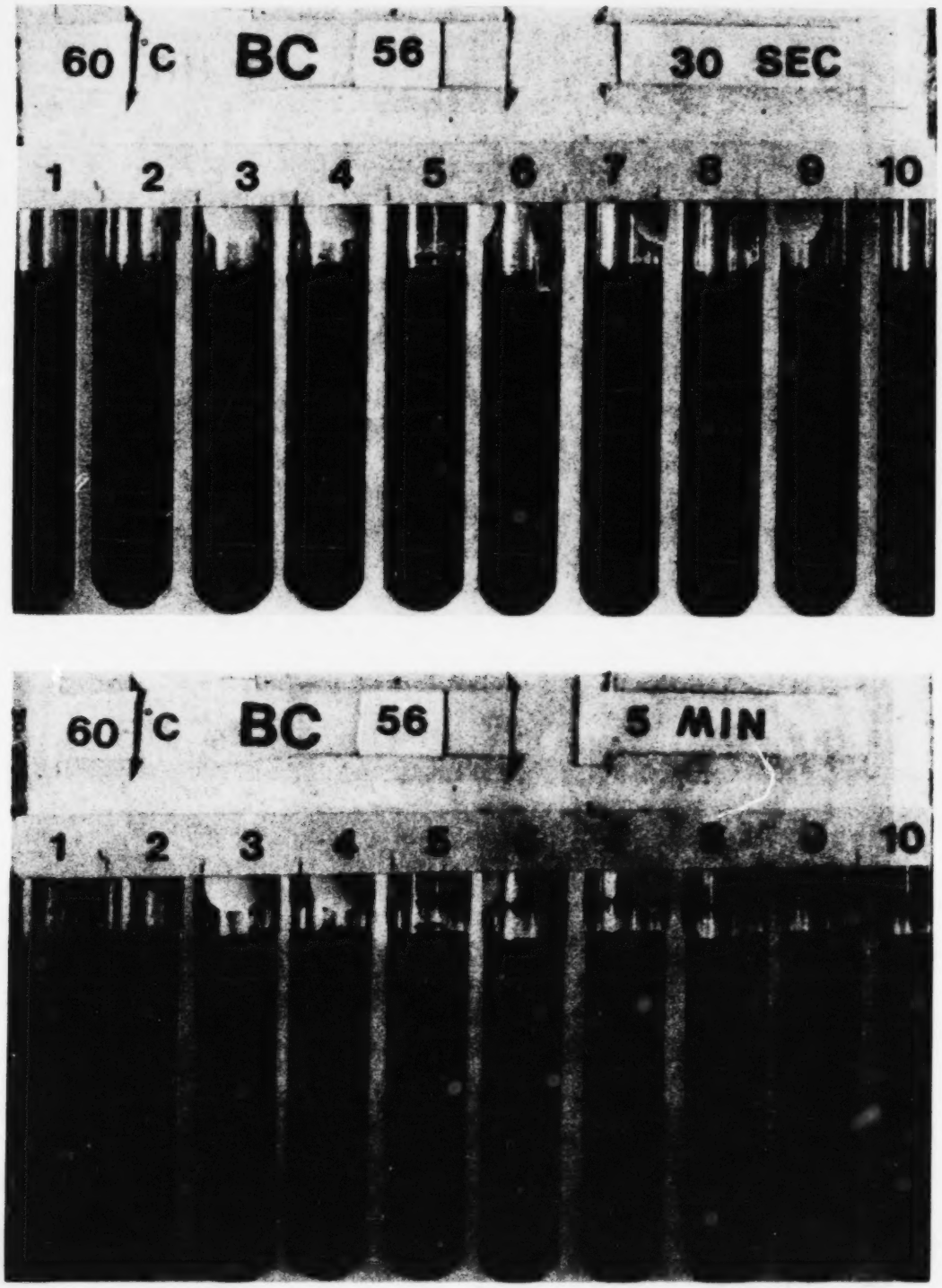
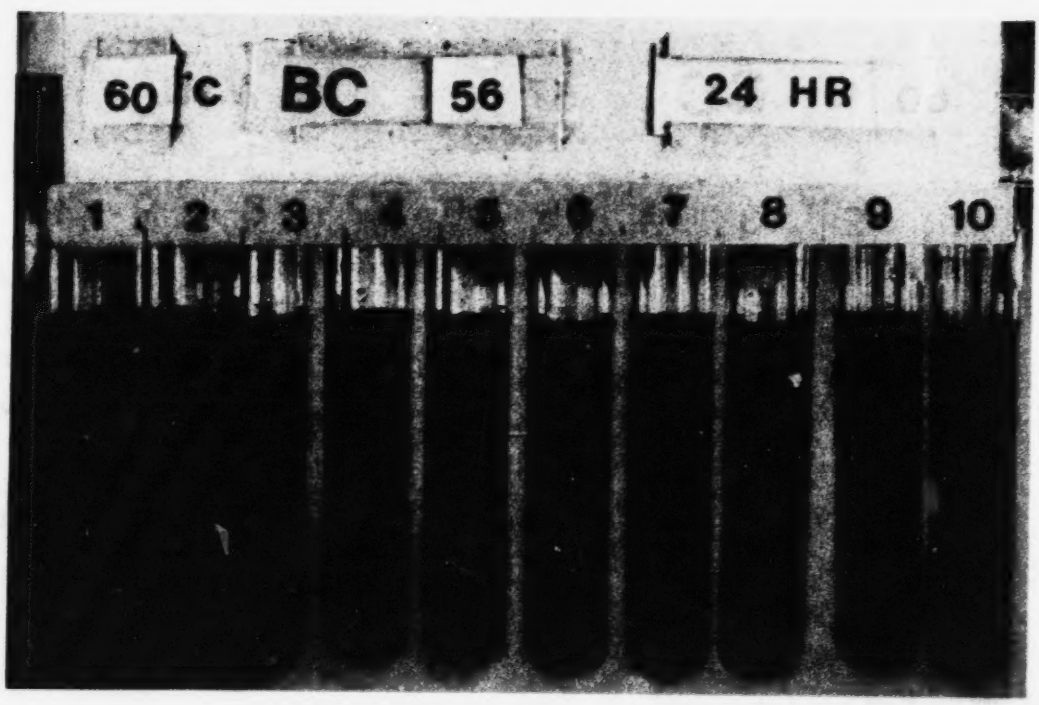


Figure 9: Concentration Optimization BC-56
#1=60 ppm, #10=150 ppm, graduated in 10 ppm steps

60 C
BC
56
1 HR
1 2 3 4 5 6 7 8 9 10


60 C
BC
56
24 HR
1 2 3 4 5 6 7 8 9 10

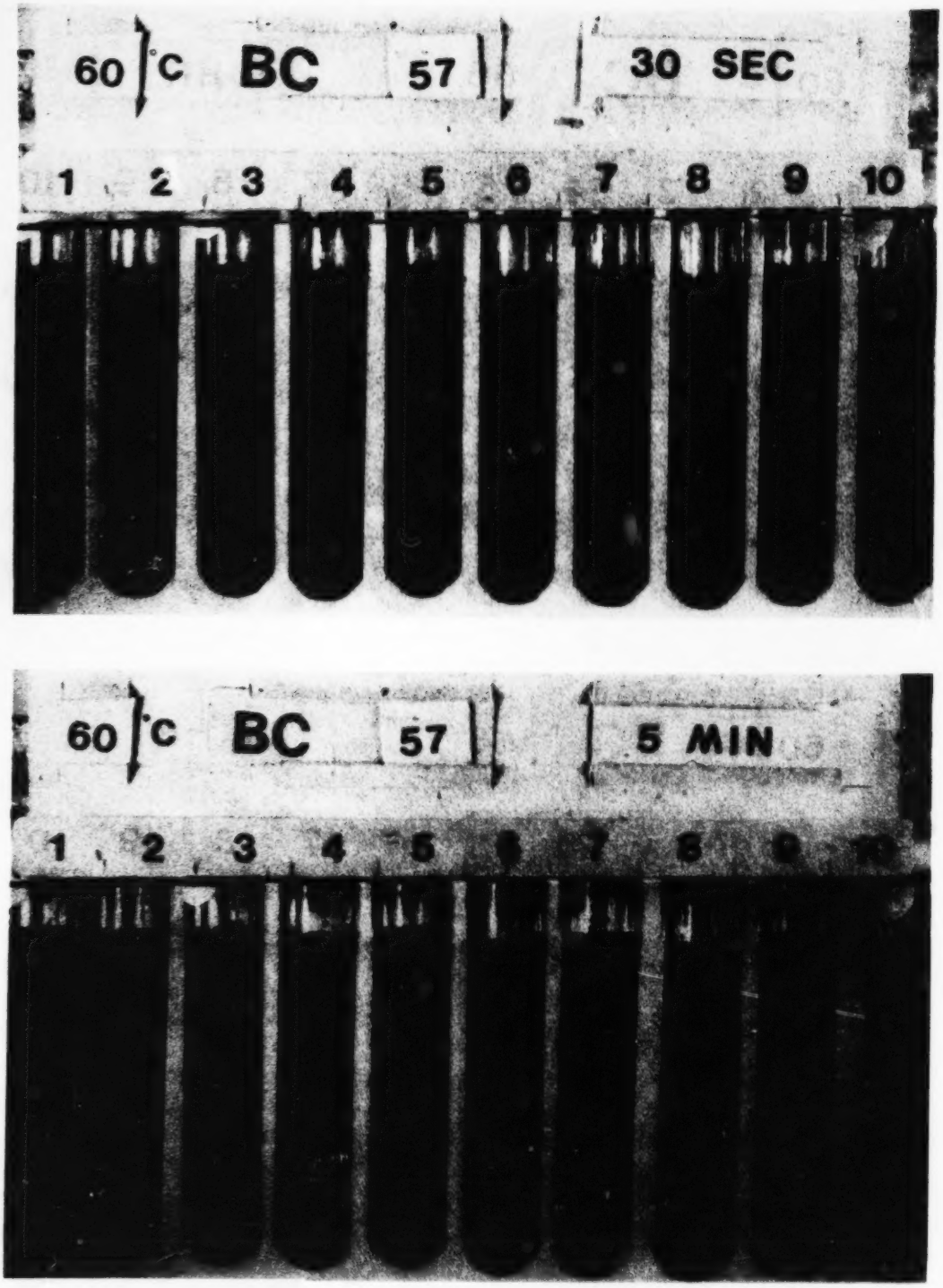
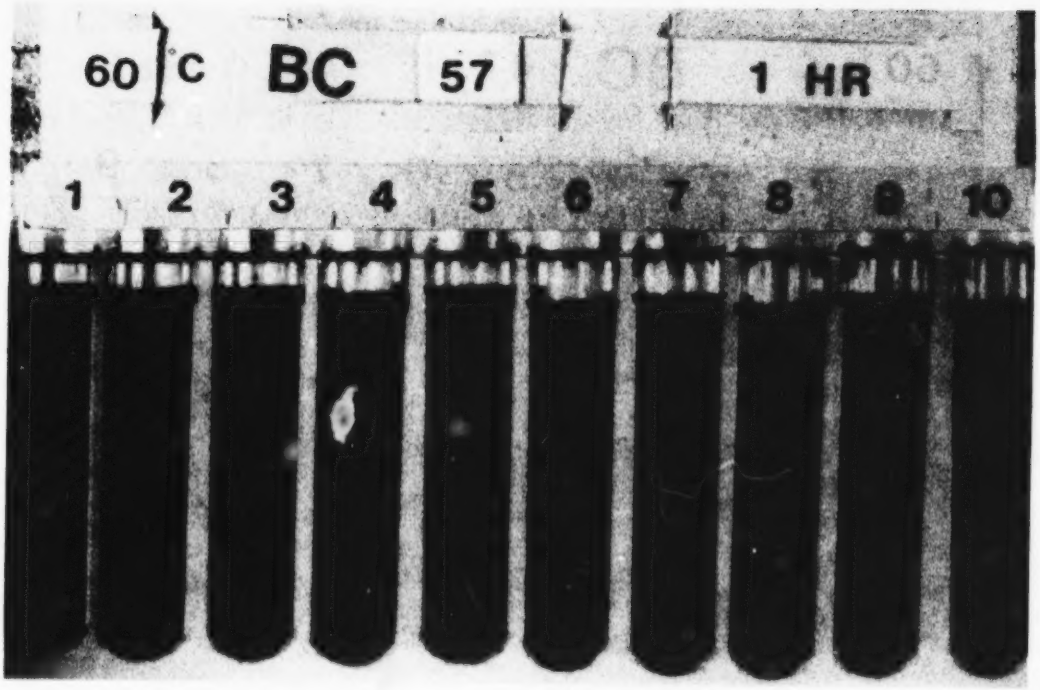
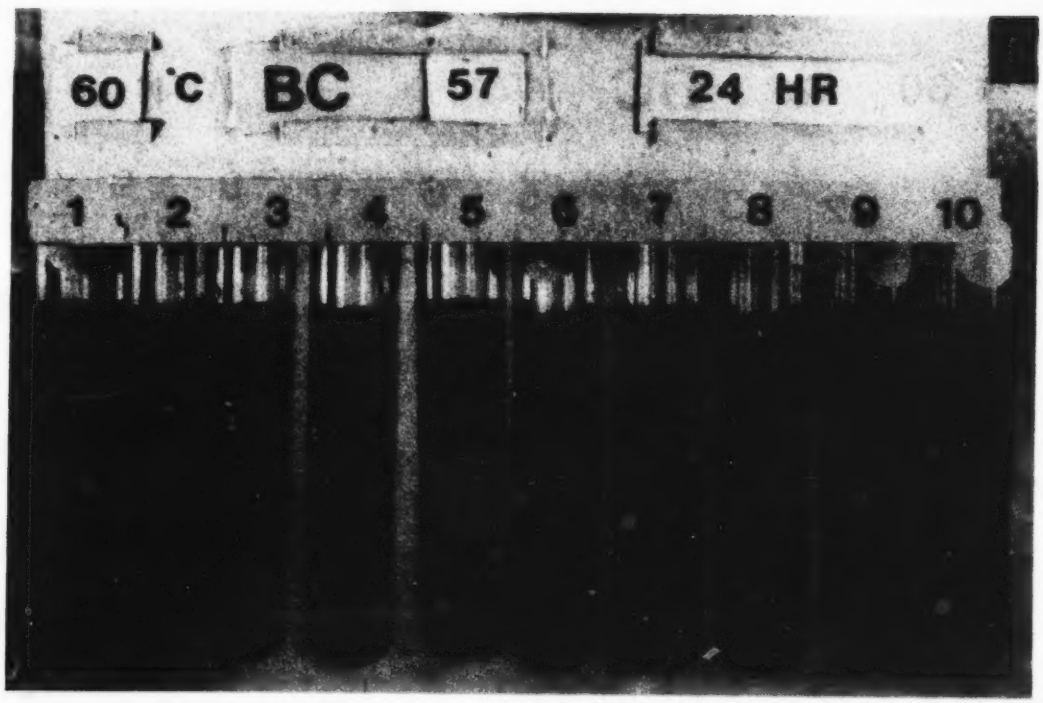


Figure 10: Concentration Optimization BC-57
#1=60 ppm, #10=150 ppm, graduated in 10 ppm steps


60 °C
BC
57
1 HR
1 2 3 4 5 6 7 8 9 10


60 °C
BC
57
24 HR
1 2 3 4 5 6 7 8 9 10

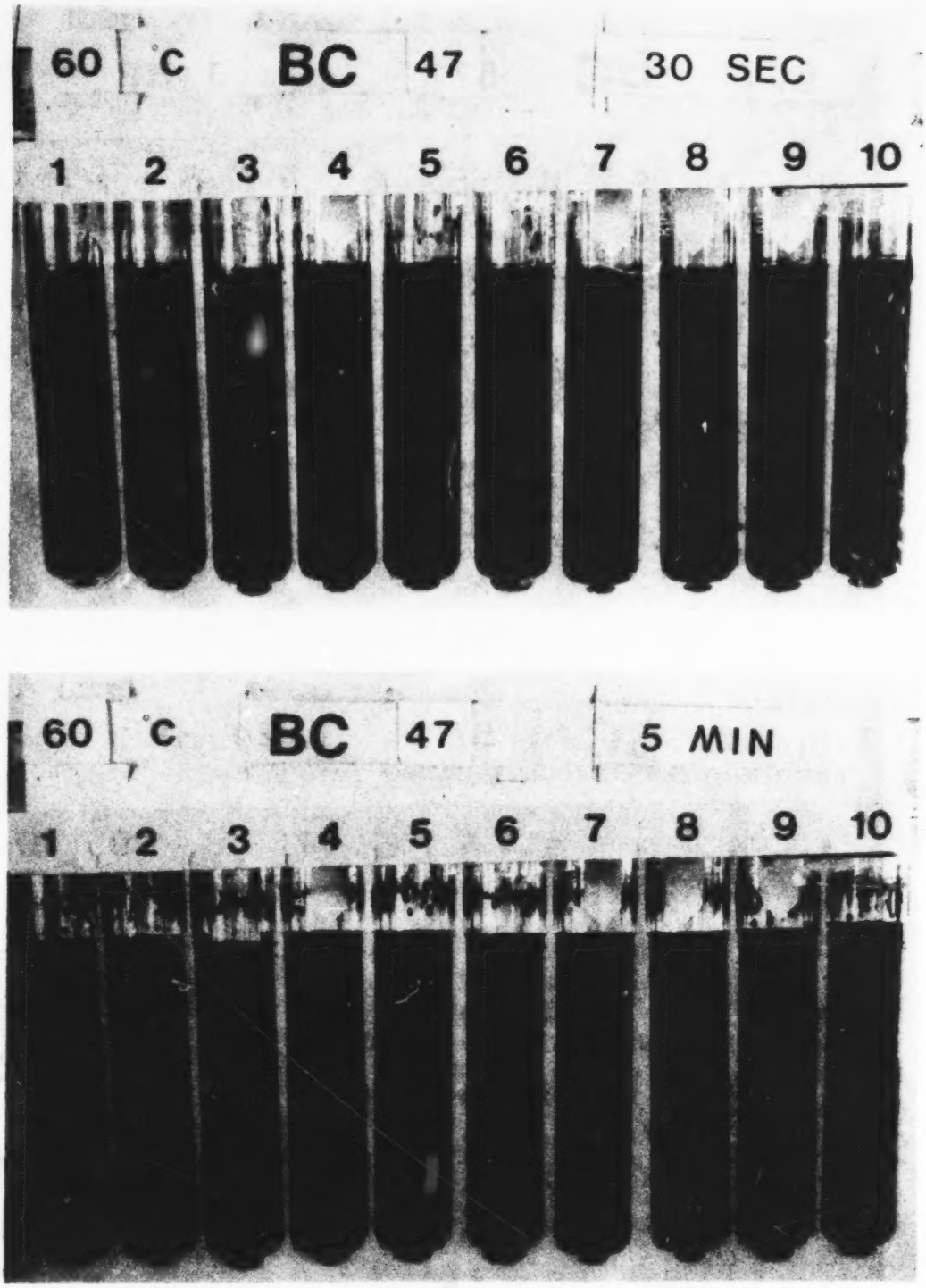


Figure 11: Concentration Optimization BC-47
#1=60 ppm, #10=150 ppm, graduated in 10 ppm steps

60 °C BC 47 1 HR
1 2 3 4 5 6 7 8 9 10

60 °C BC 47 24 HR
1 2 3 4 5 6 7 8 9 10

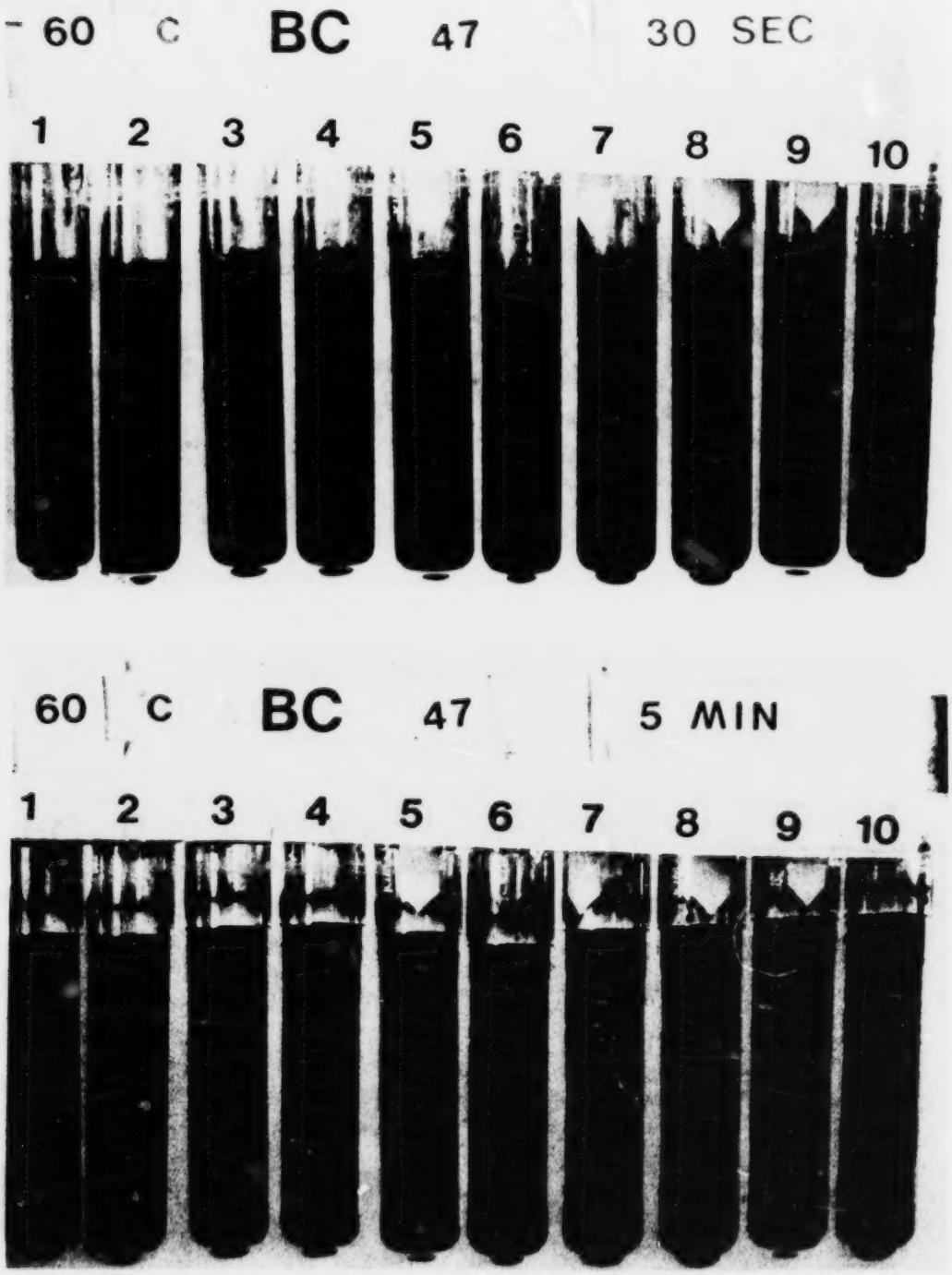
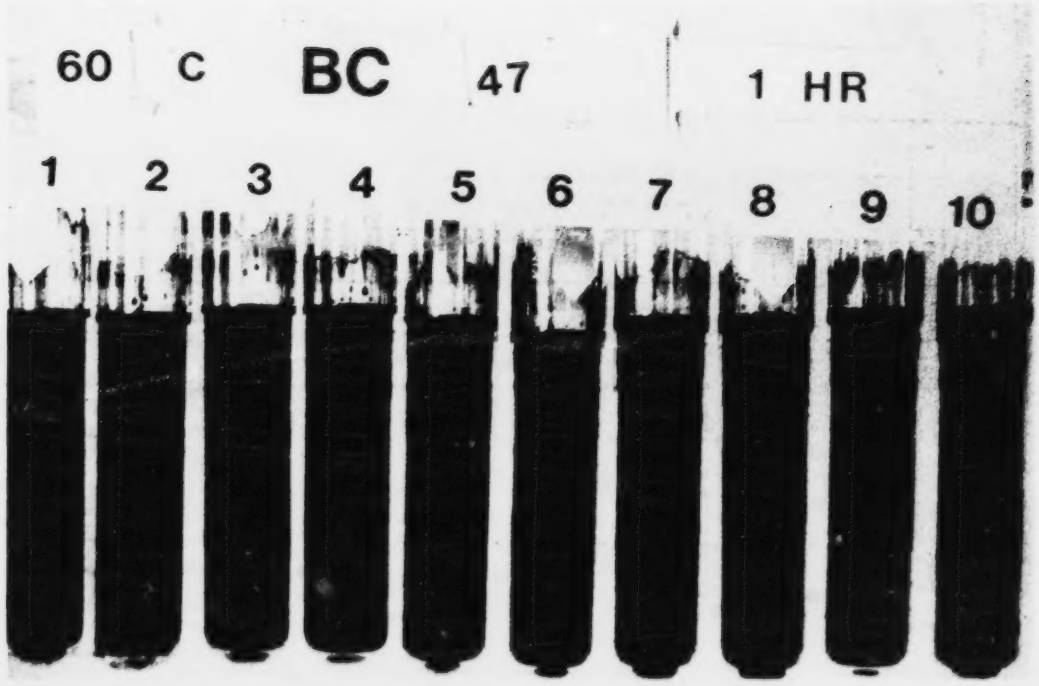
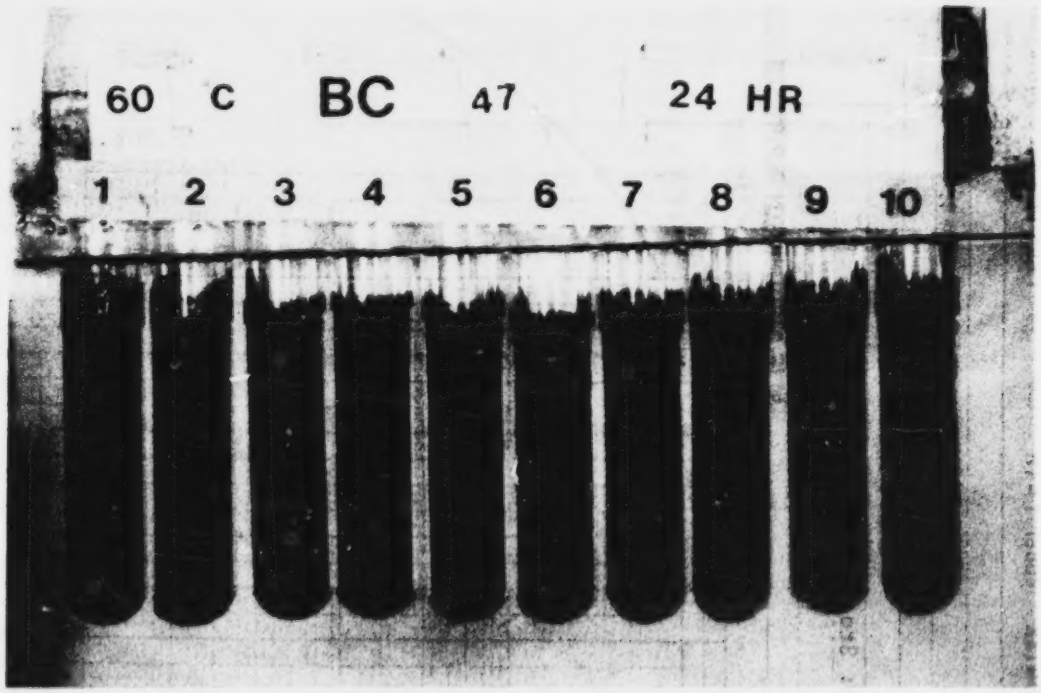


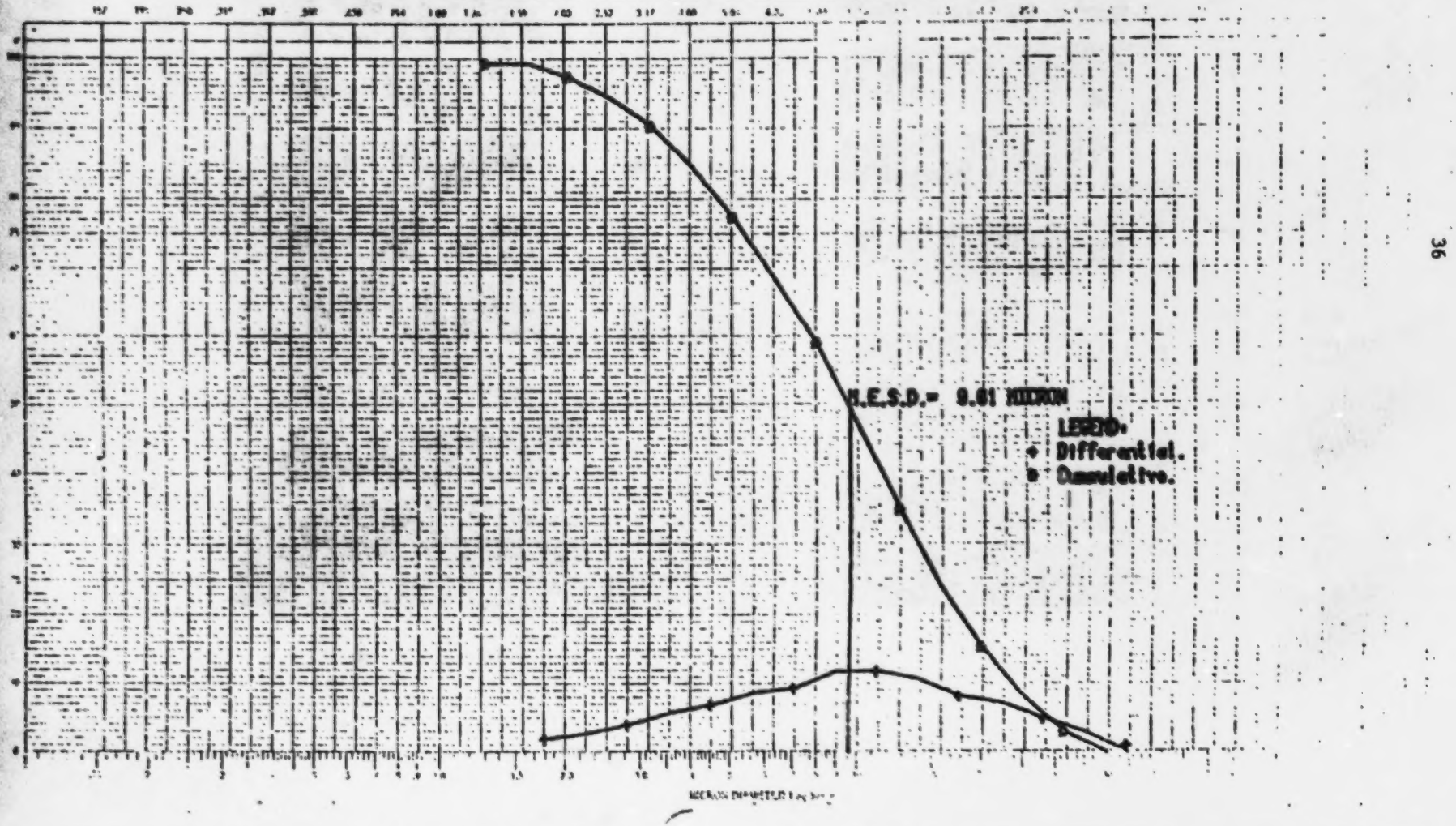
Figure 12: Concentration Optimization BC-47
#1=160 ppm, #10=250 ppm, graduated in 10 ppm steps


60 C BC 47 1 HR
1 2 3 4 5 6 7 8 9 10


60 C BC 47 24 HR
1 2 3 4 5 6 7 8 9 10

COULTER COUNTER® Model T&TA PARTICLE SIZE ANALYSIS

Figure 13: Stock Emulsion



COULTER COUNTER® Model T&TA PARTICLE SIZE ANALYSIS

ORGANIZATION
OPERATOR
EQUIPMENT

APER. SIZE | SERIAL

	SAMPLE	ELECTROLYTE	DISPERSANT
	Figure 14:BC-55, 400 ppm		

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.

M.E.S.D.= 7.40 MICRON

LEGEND:
◆ Differential.
■ Cumulative.

MICRON DIAMETER Log Scale

COULTER COUNTER® Model T & TA PARTICLE SIZE ANALYSIS

ORGANIZATION

OPERATOR

EQUIPMENT

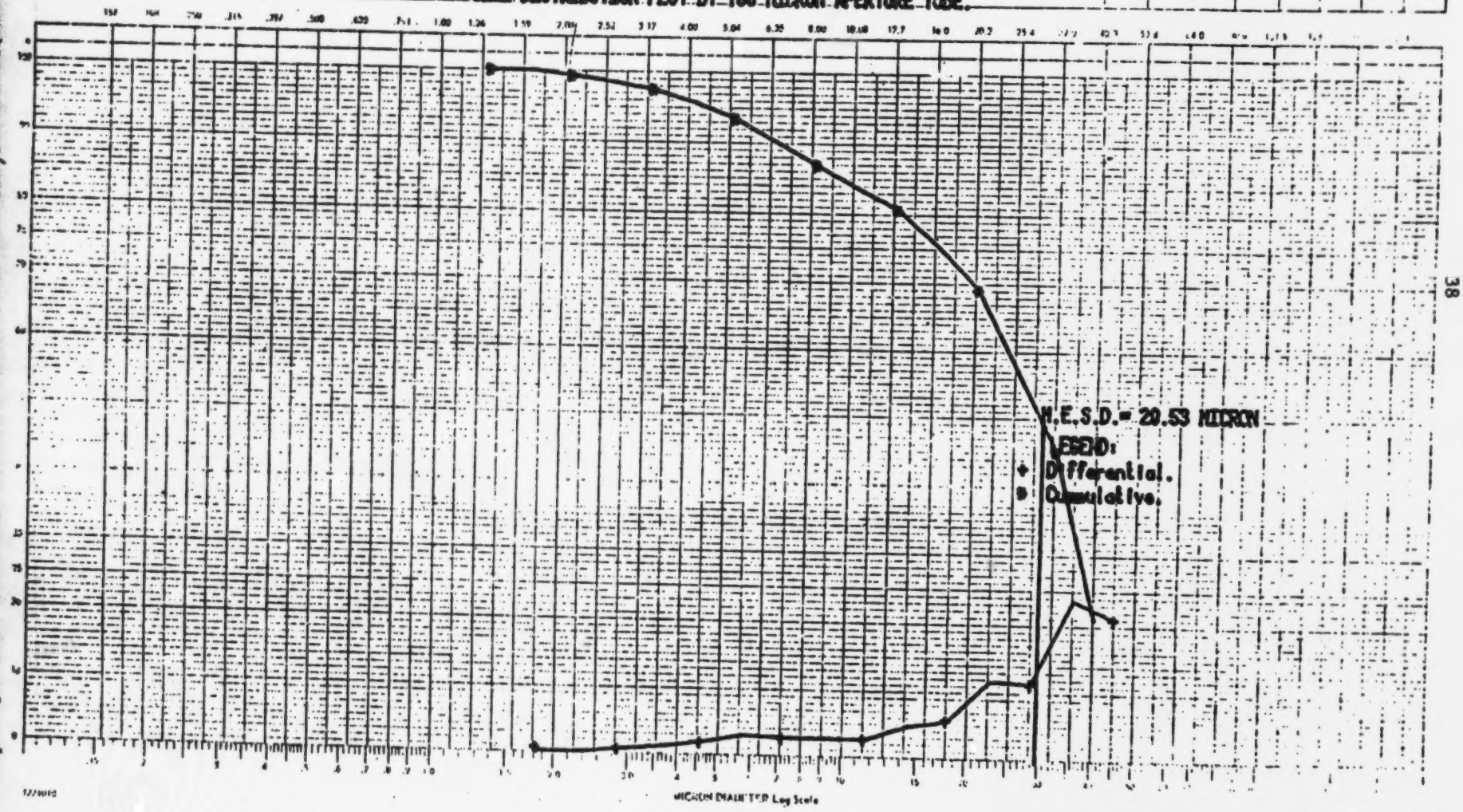
$L = d\sqrt[3]{\frac{\pi}{6}}$ $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ $\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^2$

FOR MODEL T FOR MODEL TA

SAMPLE	ELECTROLYTE	DISPERSANT	APER. SIZE	SERIAL	APT. DIA	W	± %	A	SAMPLE DETAILS D.A.	W	± %	A

Figure 15: BC-56, 200 ppm

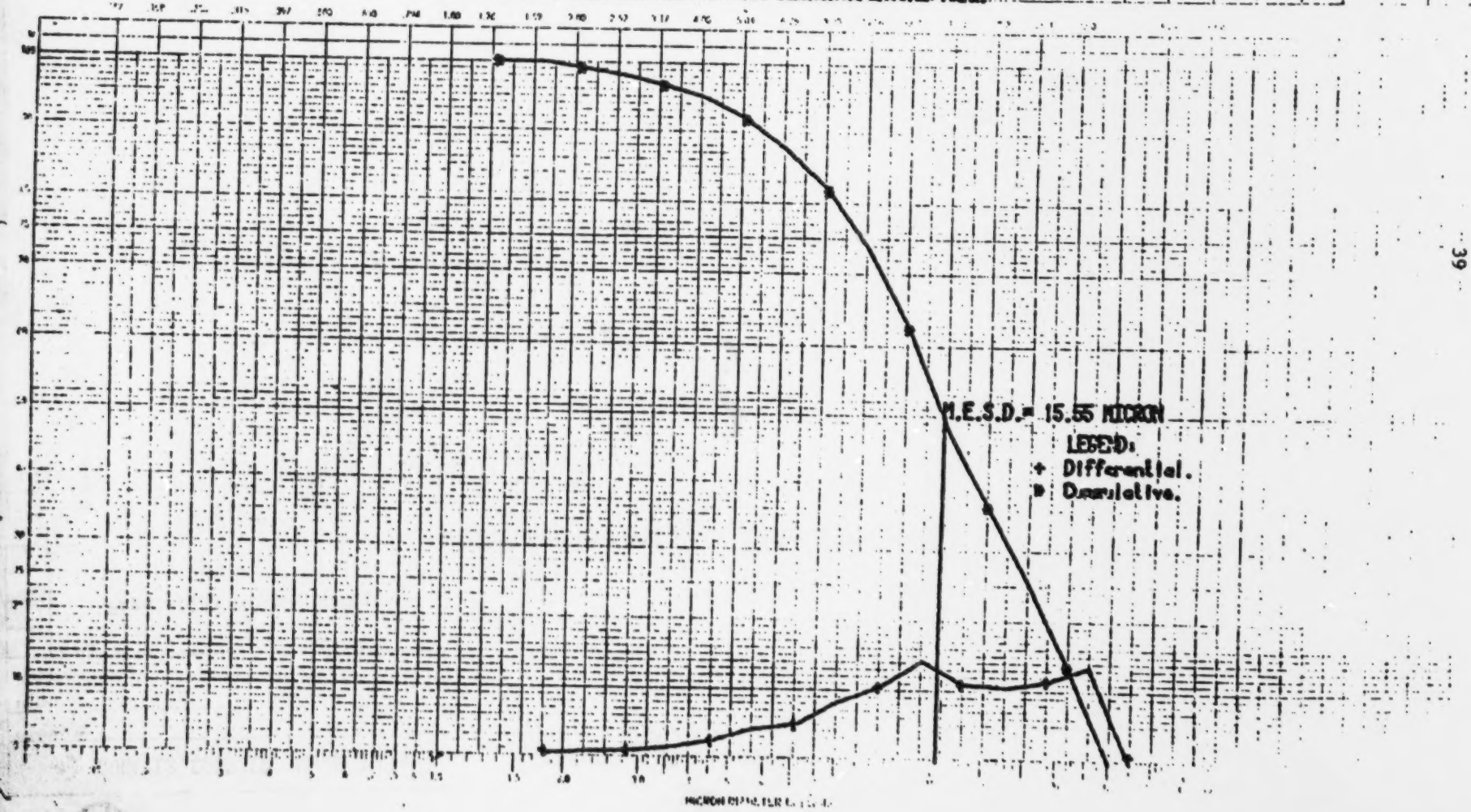
SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.



COULTER COUNTER® Model T&TA PARTICLE SIZE ANALYSIS

Figure 16: BC-56, 400 ppm

SIZE DISTRIBUTION PLOT BY 189 MICRON APERTURE TUBE.



COULTER COUNTER® Model T&TA

PARTICLE SIZE ANALYSIS

ORGANIZATION

OPERATOR

COMPANY

SAMPLE ELECTROLYTE DISPERSANT

APER. SIZE SERIAL

Figure 17: BC-57, 200 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.

M.E.S.D.= 29.44 MICRON

LEGEND:

+ Differential.

• Cumulative.

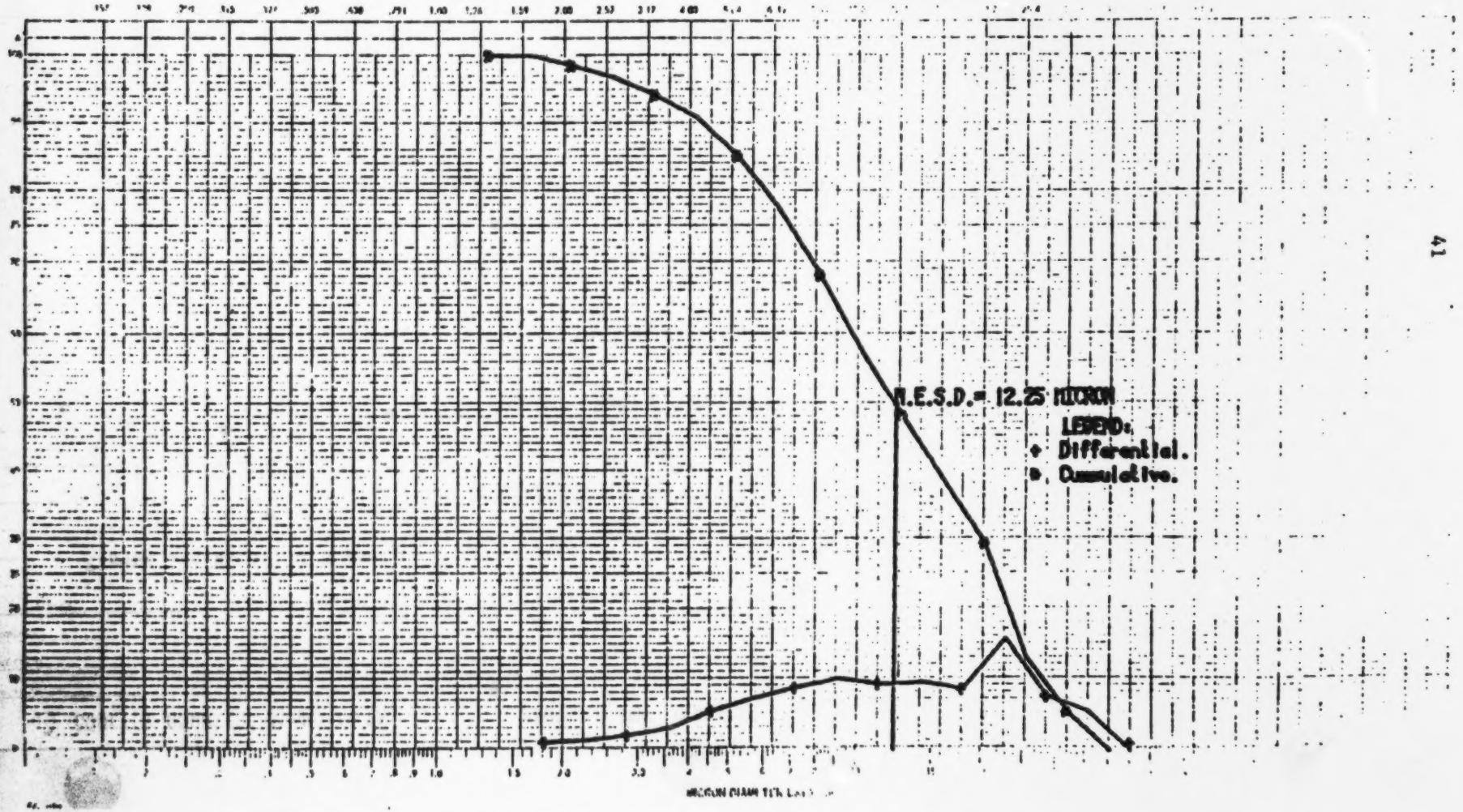
MICRON DIAMETER Log Scale

COULTER COUNTER Model T&TA PARTICLE SIZE ANALYSIS

SAMPLE ELECTROLYTE DISPERSANT

Figure 18: BC-57, 400 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.



COULTER COUNTER® Model T&TA

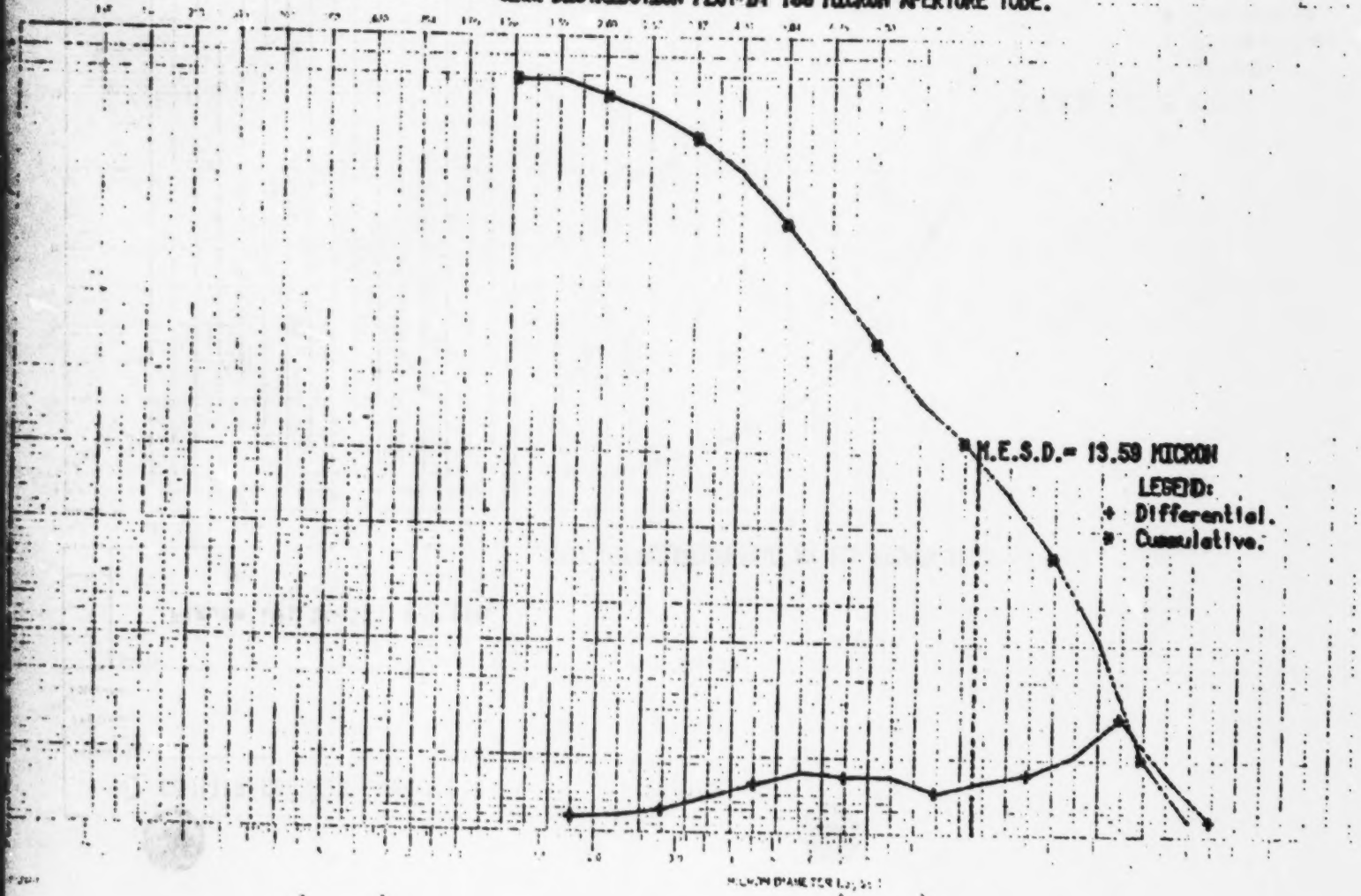
PARTICLE SIZE ANALYSIS

SAMPLE ELECTROLYTE DISPERSANT

APER. SIZE SERIAL

Figure 19: BC-47, 200 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.



COULTER COUNTER™ Model T&TA PARTICLE SIZE ANALYSIS

Figure 20: BC-47, 400 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.

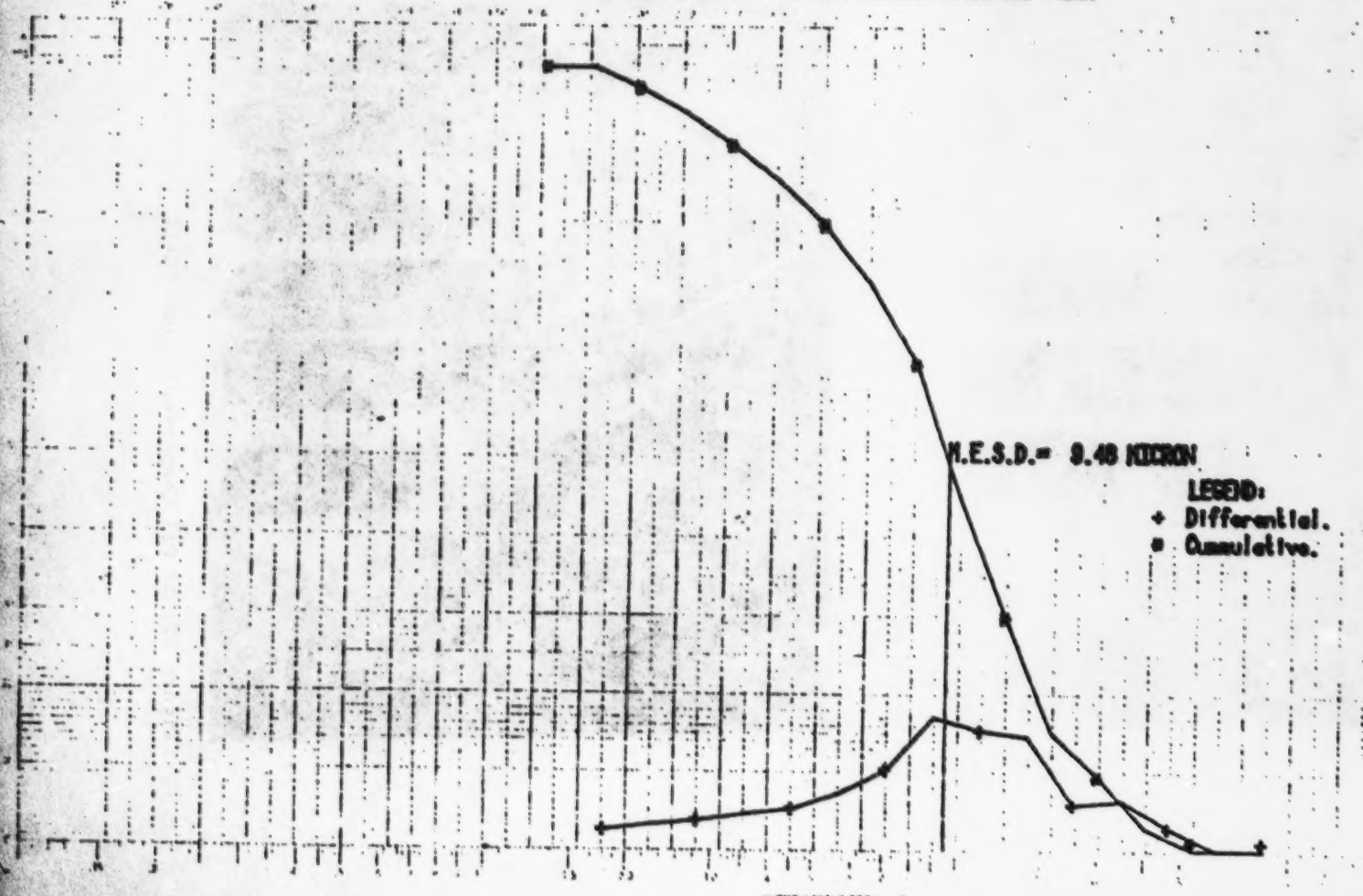


Figure 21: 5% Oil Comparison Study BC-17

Figure 22: 5% Oil Comparison Study BC-19

Figure 23: 5% Oil Comparison Study BC-21

Figure 24: 5% Oil Comparison Study BC-22

Figure 25: 5% Oil Comparison Study BC-47

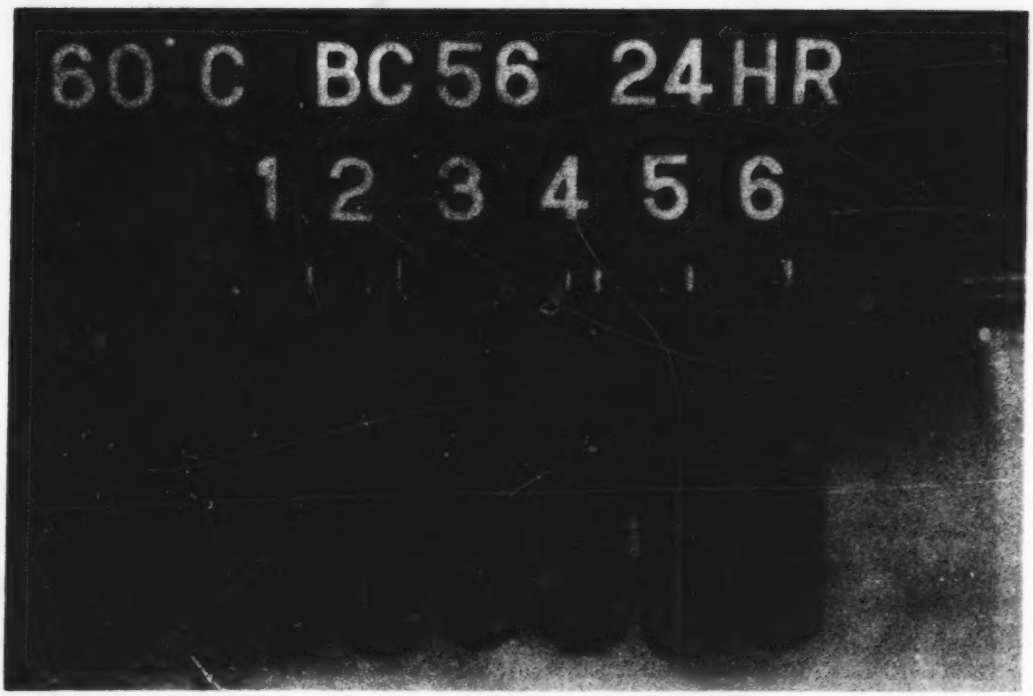


Figure 26: 5% Oil Comparison Study BC-56

Figure 27: 5% Oil Comparison Study BC-57

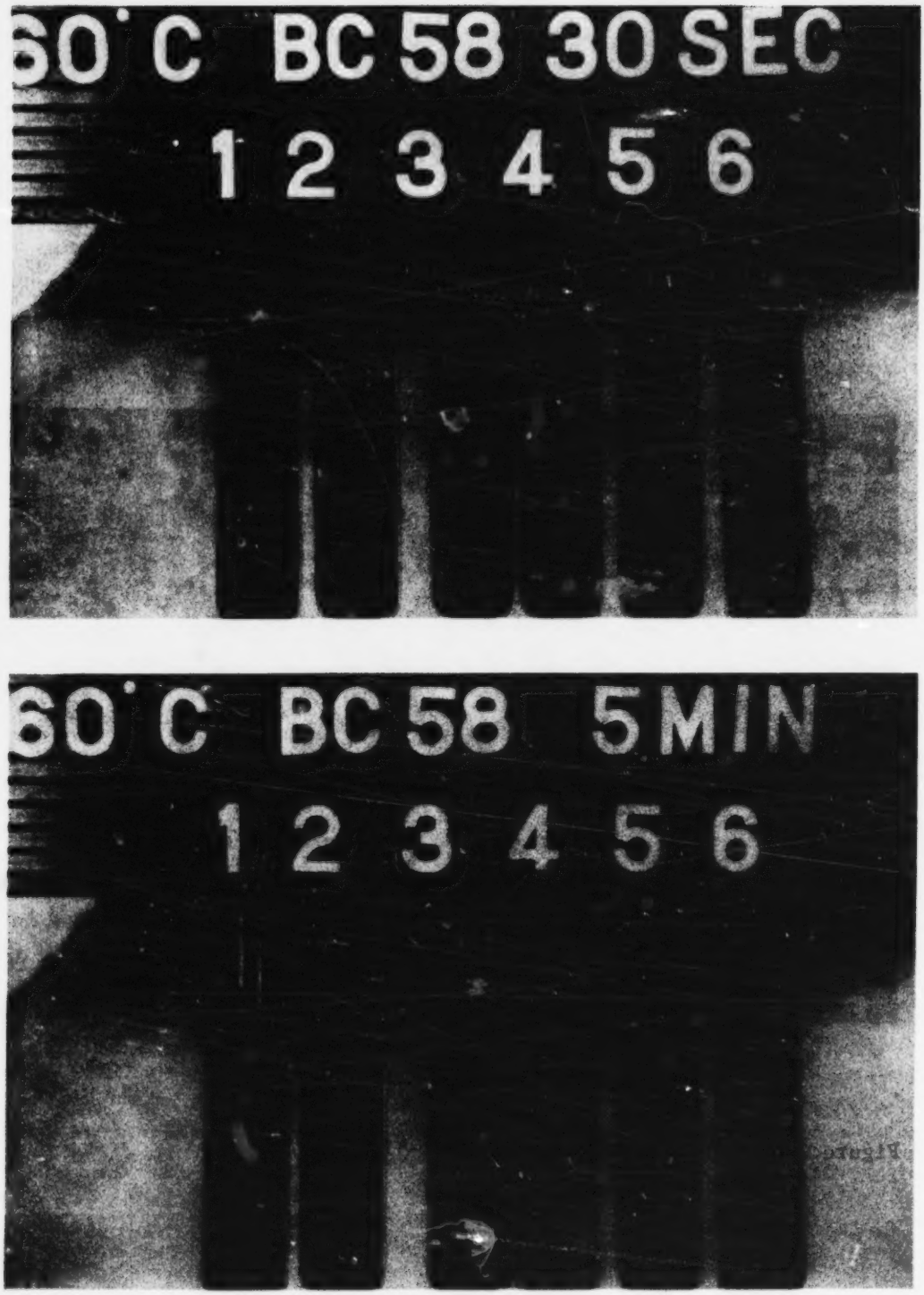
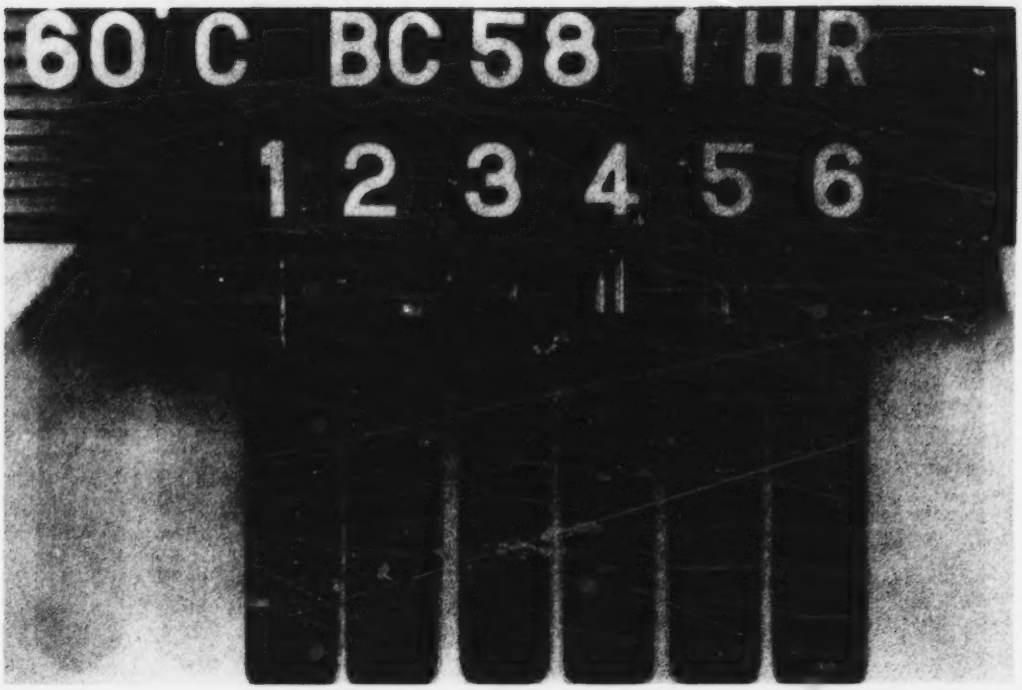
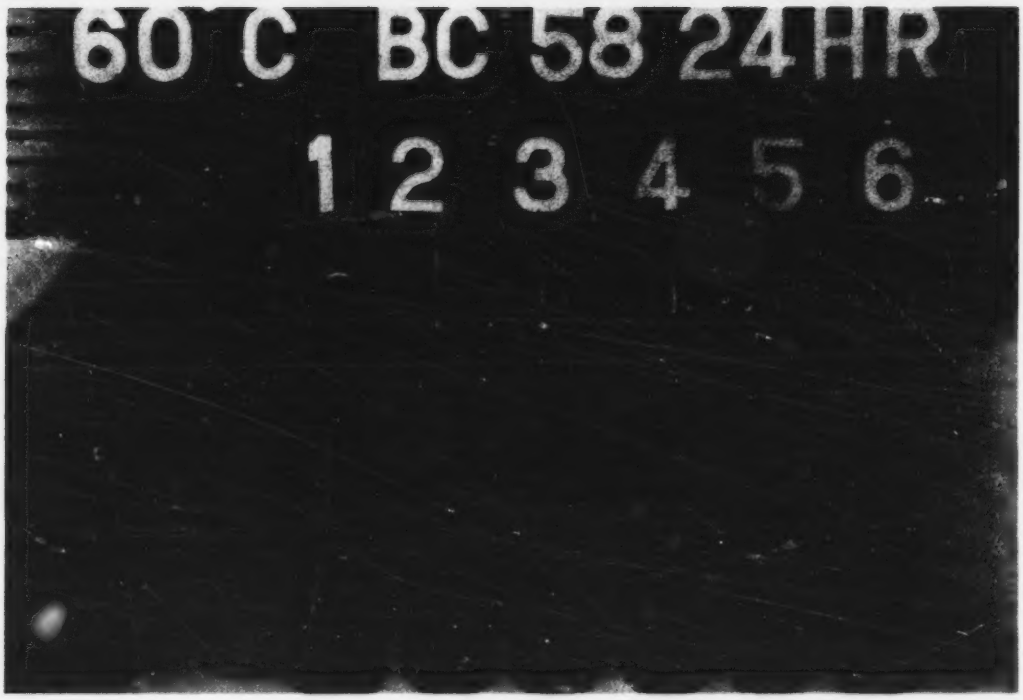


Figure 28: Initial Screening BC-58


60 C BC 58 1 HR
1 2 3 4 5 6


60 C BC 58 24 HR
1 2 3 4 5 6

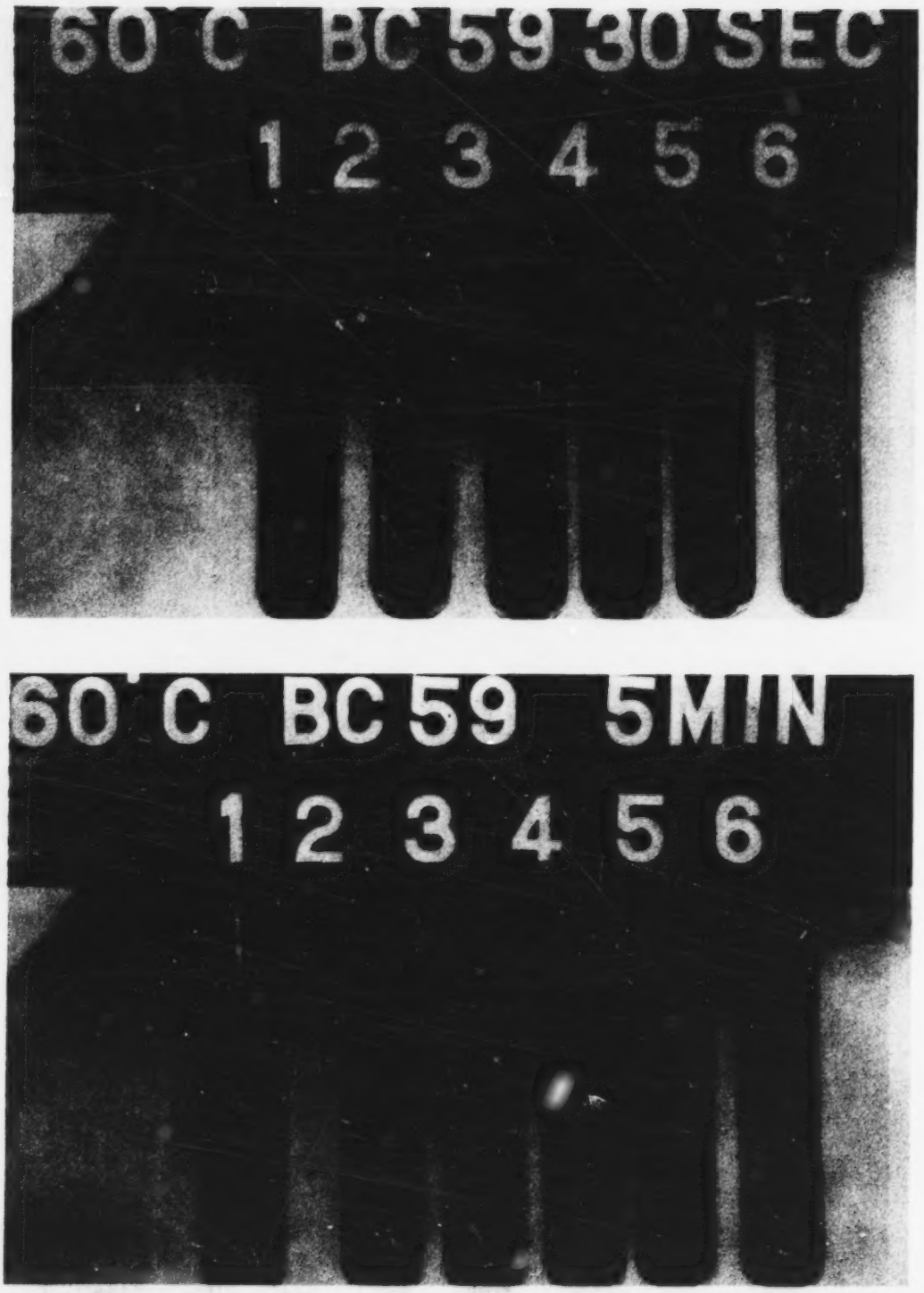


Figure 29: Initial Screening BC-59

60 C BC 59 1 HR
1 2 3 4 5 6

60 C BC 59 24 HR
1 2 3 4 5 6

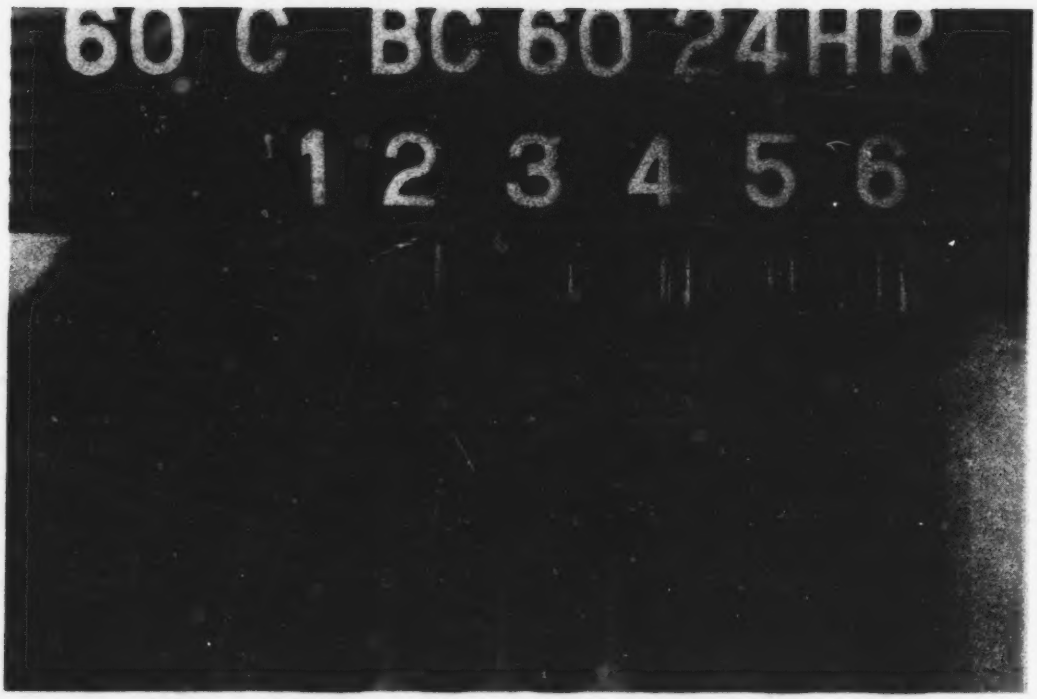
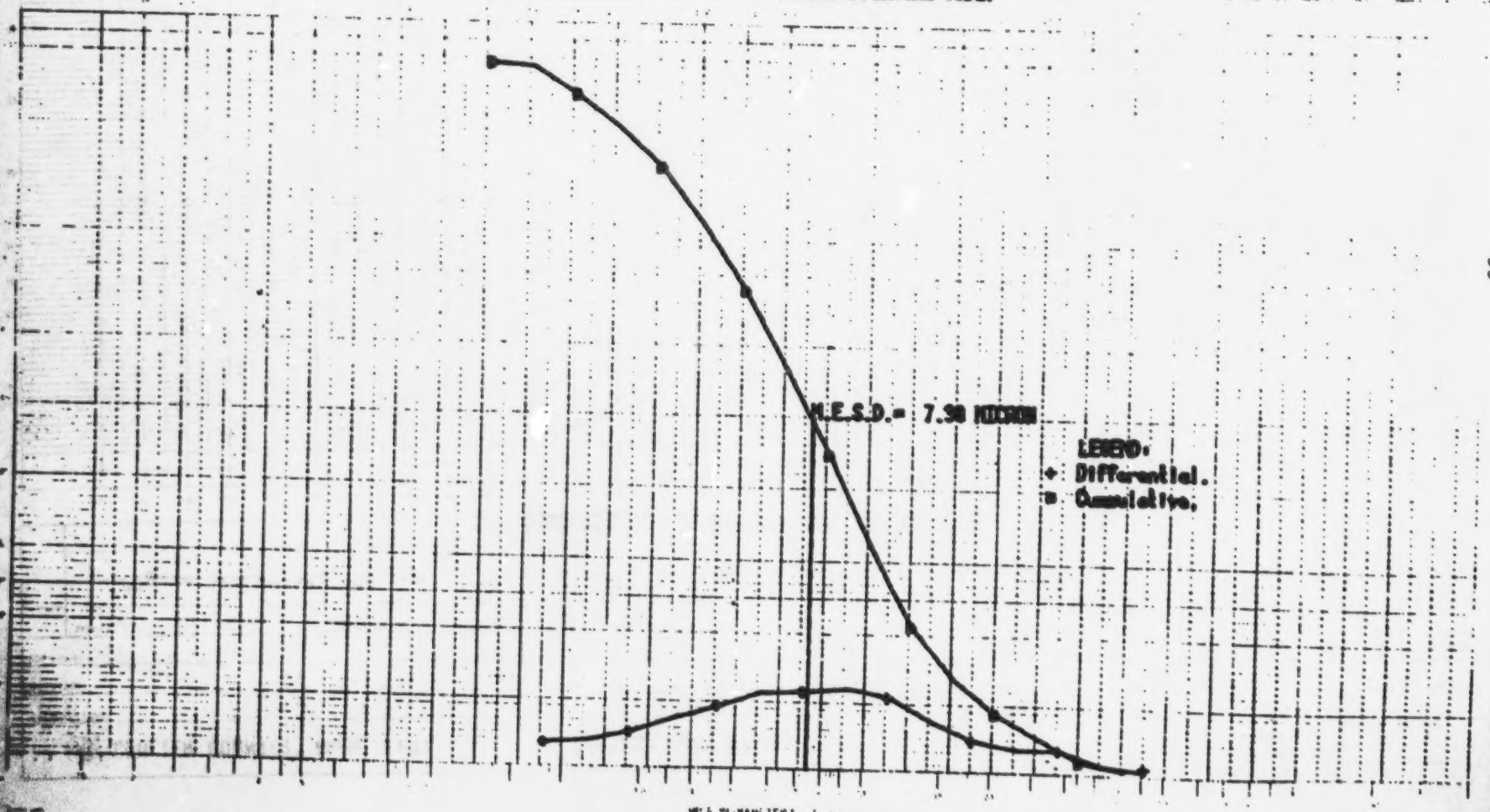


Figure 30: Initial Screening BC-60

COULTER COUNTER' Model T&TA

PARTICLE SIZE ANALYSIS

Figure 31: Stock Emulsion



COULTER COUNTER® Model T&TA

PARTICLE SIZE ANALYSIS

.10 – 200 μ
2 PERCENT

COULTER ELECTRONICS
200 W 20 ST.
HIALEAH FLA. 33010

ORGANIZATION

OPERATOR

EQUIPMENT

$d^{new} = d\sqrt[3]{\frac{T}{T'}}$

FOR MODEL T

$\frac{\Delta q}{\Delta i} = \left(\frac{q_2}{i_1}\right)^2$ when $q_2 = i_1$

$\frac{\Delta i}{\Delta i} = \left(\frac{i_1}{i_2}\right)^3$ when $i_2 = i_1$

FOR MODEL TA

SAMPLE	ELECTROLYTE	DISPERSANT	APER. SIZE	SERIAL		PART DIA.	M	± SA	A	SAMPLE SETTINGS DIA.	M	± SA	A

Figure 32: BC-58, 400 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.

.157 .198 .250 .315 .397 .500 .630 .794 1.00 1.26 1.59 2.00 2.52 3.17 4.00 5.04 6.35 8.00 10.08 12.7 16.0 20.2 25.4 32.0 40.3 50.8 64.0 80.6 101.6 128.0 161.3 203.2 256.0 322.5

M.E.S.D.= 14.12 MICRON

LEGEND:
• Differential
• Cumulative

MICRON DIAMETER Log Scale

COULTER COUNTER® Model T&TA

PARTICLE SIZE ANALYSIS

.15 - 280 μ
1 PERCENT

COULTER ELECTRONICS INC.
590 W 20 ST.
HIALEAH FLA. 33010

ORGANIZATION
OPERATOR
EQUIPMENT

$k = d\sqrt[3]{\frac{\pi}{6}}$ FOR MODEL T

$\frac{A_2}{A_1} = \left(\frac{d_2}{d_1}\right)^3$ when $V_2 = V_1$ FOR MODEL TA

$\frac{A_2}{A_1} = \left(\frac{d_1}{d_2}\right)^3$ when $V_2 = V_1$

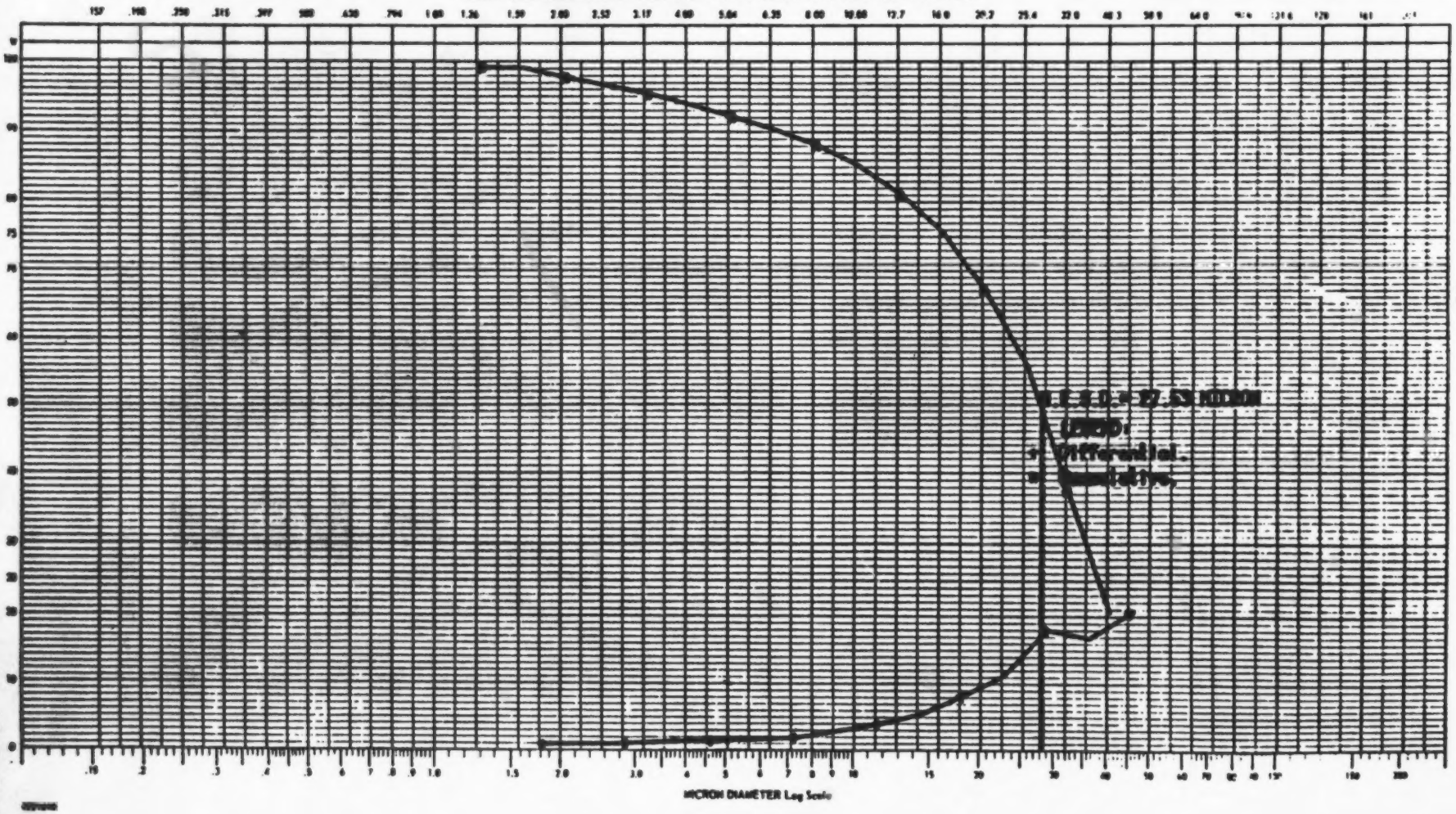
SAMPLE	ELECTROLYTE	DISPERSANT	APER. SIZE	SERIAL		PART DIA.	V	± DA	A

SAMPLE SETTINGS

DIA.	V	± DA	A

Figure 33: BC-59, 400 ppm

SIZE DISTRIBUTION PLOT BY 100 MICRON APERTURE TUBE.



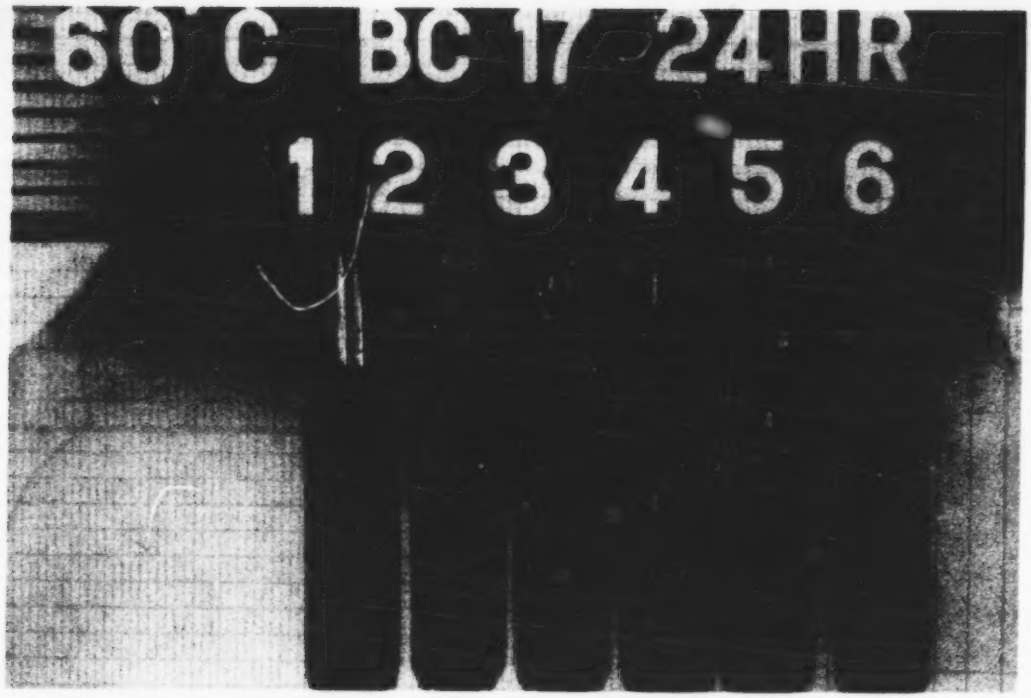


Figure 34: 7% Oil Comparison Study BC-17

Figure 35: 7% Oil Comparison Study BC-19

Figure 36: 7% Oil Comparison Study BC-21

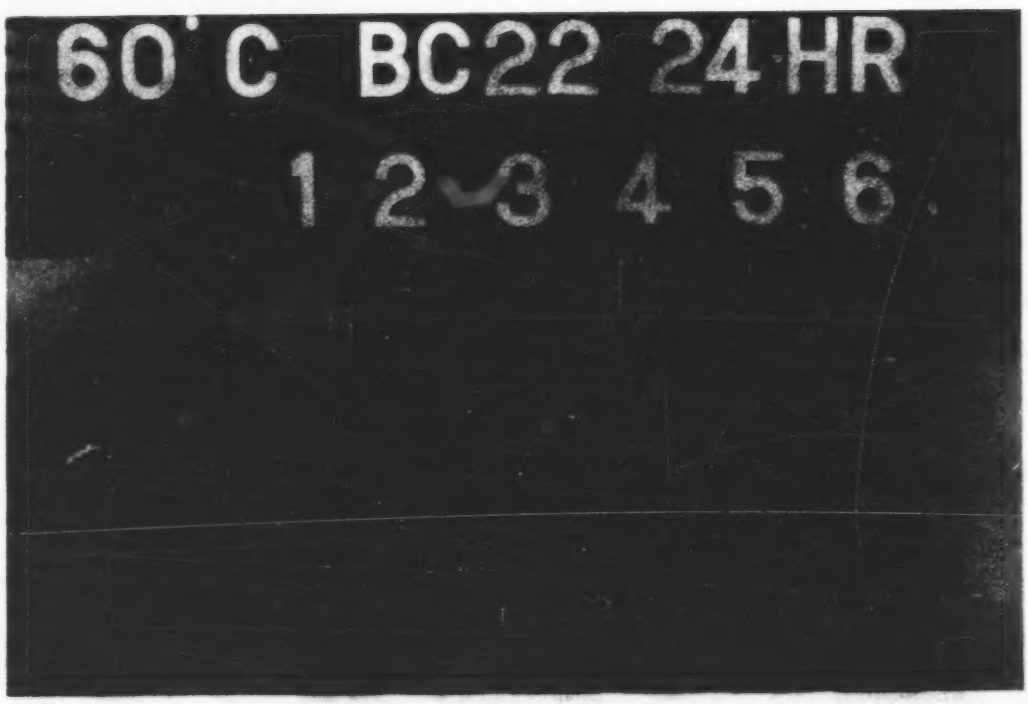


Figure 37: 7% Oil Comparison Study BC-22

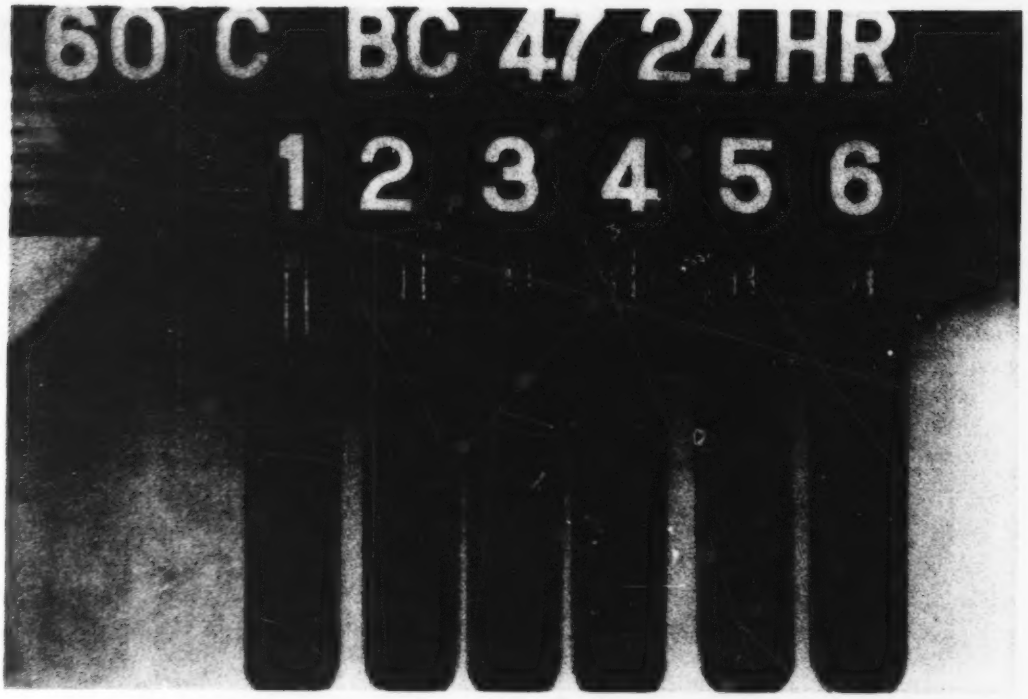


Figure 38: 7% Oil Comparison Study BC-47

Figure 39: 7% Oil Comparison Study BC-56

Figure 40: 7% Oil Comparison Study BC-57

Figure 41: 3% Oil Comparison Study BC-17

Figure 42: 3% Oil Comparison Study BC-19

Figure 43: 3% Oil Comparison Study BC-21

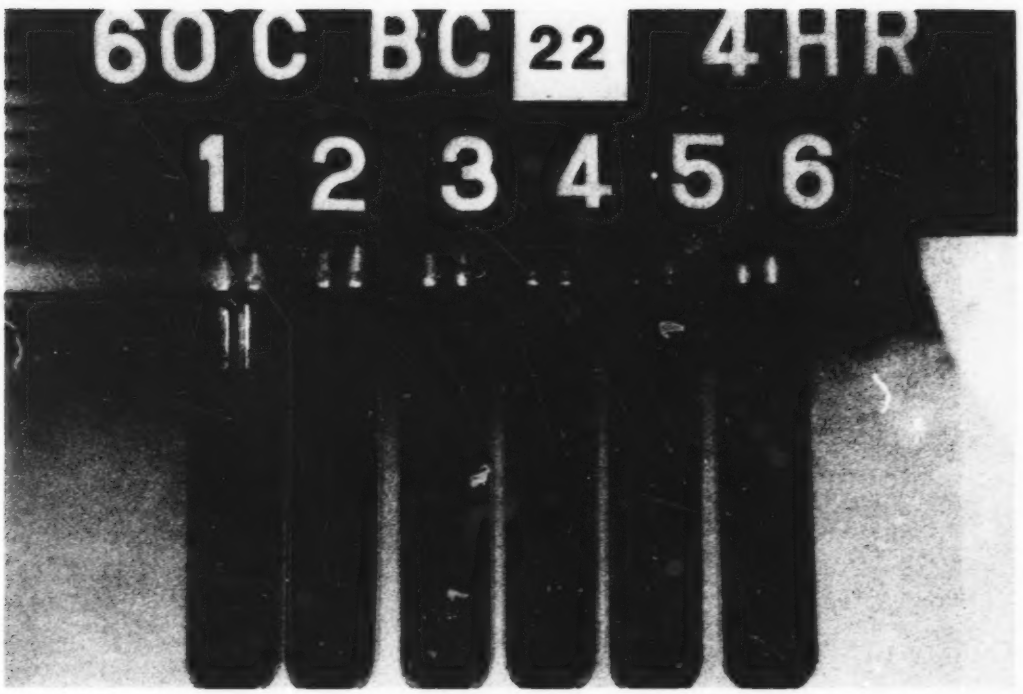


Figure 44: 3% Oil Comparison Study BC-22

Figure 45: 3% Oil Comparison Study BC-47

Figure 46: 3% Oil Comparison Study BC-56

Figure 47: 3% Oil Comparison Study BC-57

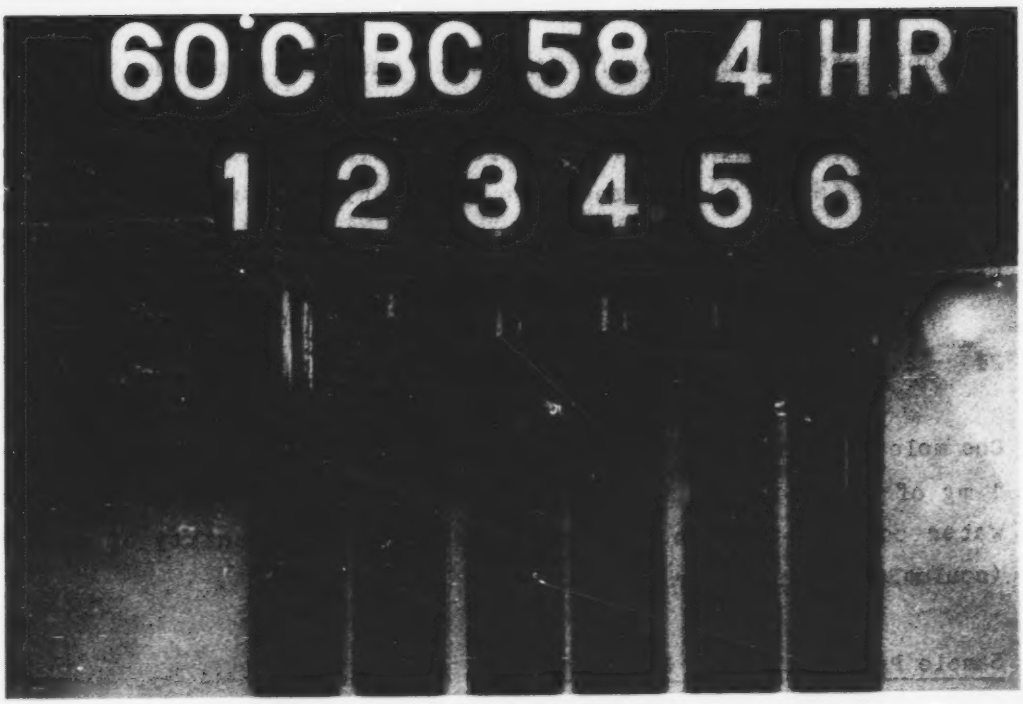


Figure 48: 3% Oil Comparison Study BC-58

APPENDIX A ANALYTICAL PROCEDURES

MOISTURE DETERMINATION

The Mitsubishi Moisture Meter model CA-02 combines coulometry with the Karl Fischer titration method. Water is titrated by the electrolytic generation of a Karl Fischer reagent which reacts with water as follows:

$$I_2+SO_2+3C_5H_5N+H_2O \text{ -------> } 2C_5H_5N.HI+C_5H_5N.SO_3 \qquad (1)$$

$$C_5H_5N.SO_3+CH_3OH \text{ ---------> } C_5H_5N.HSO_4CH3 \qquad (2)$$

In Coulometry-Karl Fischer titration, the sample is added to a pyridine-methanol solution (with iodide ion and sulphur dioxide as principal components). Iodine, generated electrolytically at the anode, reacts with the water in the sample as shown in equations (1) and (2). Iodine is generated in direct proportion to the quantity of electricity according to Faraday's Law. For example:

$$2I^- - 2e^- \text{ ------> } I_2 \qquad (3)$$

One mole of iodine reacts quantitatively with one mole of water. Therefore, 1 mg of water is equivalent to 10.71 coulombs. Based on this principle, water content is directly determined from the quantity of electricity (coulombs) required for electrolysis.

Sample Preparation and Procedures

The samples to be analyzed are sufficiently diluted with tetrahydrofuran so that the final concentration for water is within the range of 10 µg to 20 mg H_2O, per injection. The diluted sample is drawn into a preweighed syringe and the sample and syringe are weighed. A known volume of sample is then injected into the titration vessel. The weight of sample injected is then determined by weighing the empty syringe. The digital readout gives the micrograms of water contained in the injected sample. By applying dilution factors and subtracting the amount of water contained in a THF blank, the percentage of water in the sample can be calculated as follows:

$$\%H_2O = \frac{(H_2O \text{ in sample} - H_2O \text{ in blank}) \ (\mu g) \times \text{dilution factor(s)} \times 10^{-4}}{\text{wt injected sample (g)}}$$

COULTER COUNTER ANALYSIS

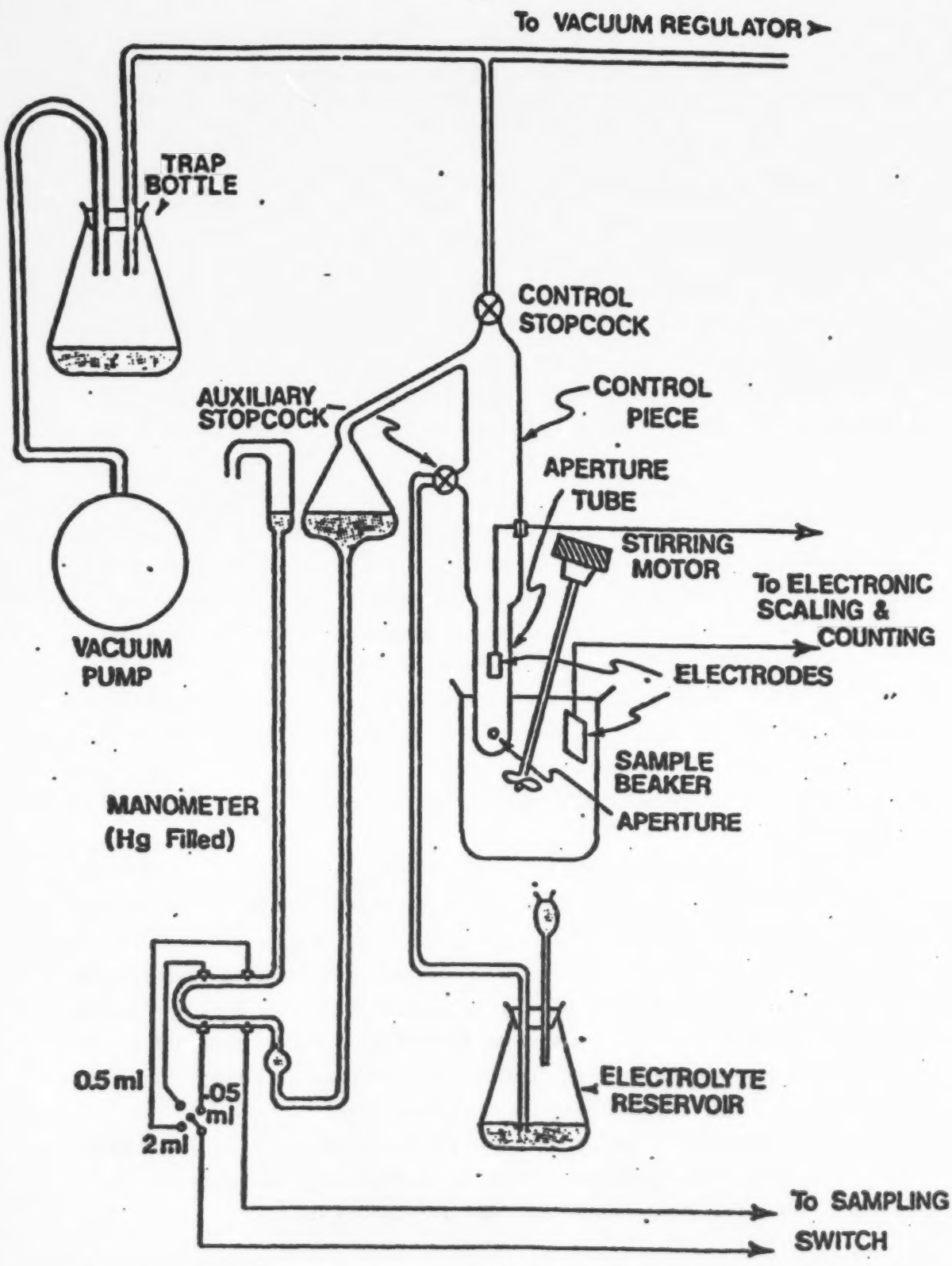
Principle

The principle of operation, referred to as the Coulter principle or the electronic sensing zone method, may be summarized as follows (see attached schematic).

The particles to be analyzed are suspended in a conductive liquid (electrolyte) and the suspension is forced by vacuum to flow through a small aperture in a glass tube. Electrodes are immersed in the conducting liquid on opposite sides of the aperture and a current passed between them. As the current must pass through the aperture, the concurrent passage of a particle will change the resistance between the electrodes. This produces a current pulse of short duration whose magnitude is, within limits, proportional to the particle volume. Current change pulses resulting from the passage of particles through the aperture are electronically converted into voltage pulses, which are amplified and then sized into 16 discrete channels by means of threshold circuits. Cumulative, as well as differential, volume data may be obtained. The number of pulses (particles) in each size range (channel) is also recorded. All data may be obtained via a numeric readout or tape printer.

Apertures are available in sizes ranging from 15 μm to 2000 μm. The useful range of an aperture tube is from approximately 2% to 40% of the aperture diameter. The current change induced by the passage of particles with diameters less than 2% of the aperture diameter becomes too small to be reliably detected and may be masked by electronic noise and interference effects. The response of the instrument to particles larger than 40% of the aperture diameter begins to significantly deviate from linearity; i.e., the response is no longer proportional to particle volume.

Each aperture tube is calibrated using standards of known size. Synthetic polymer latex particles, which are spherical, low-density, non-porous and essentially mono-sized are most frequently used.



The Coulter Counter: A Schematic of the Particle Sensing System.

The conducting liquid (electrolyte) must be chosen to meet the conductivity requirements of the aperture and should not react in any way with the particulate system to be analysed. The current flow through the aperture is adjustable; the optimum value depends on the size of the aperture and the conductivity of the electrolyte used. Proper aperture current is necessary to obtain maximum signal to noise ratio during operation. In our laboratory, noise generated as a result of external interferences was minimized by construction of a Faraday cage around the sample stand.

As previously stated the Coulter Counter sizes each pulse (particle) into one of sixteen channels; the assignment of particle size to channel being accomplished by electronic thresholds built into the instrument. A regular geometric size progression exists throughout the sixteen channels. Each channel accumulates particles within 2:1 volumetric size range or a diameter range of 3/2. A three-channel span therefore represents a diameter change of 2:1. The amount of sample analyzed is determined by presenting (1) the time (duration) of the count, (2) the number of particles to be counted, or (3) the volume of sample on which the accumulation is to be made. Sufficient data should be obtained to allow the instrument to normalize the size distribution to 100%. Time required for one accumulation is 1-2 min; normally three accumulations are made and the average taken.

Differential and cumulative volume per cent data for each channel (size range) are typically plotted. Median particle (droplet) size known as MESD (Median Equivalent Spherical Diameter) is conveniently taken as the 50% point on the cumulative curve.

Sample Preparation and Procedure

The samples measured with the Coulter Counter are mostly aqueous and the particulates to be measured (whether as solids or oil) are the minor component. These samples must be pre-filtered to remove materials too large for the size of aperture in use. For example, when using a 100 μm aperture, the samples are pre-filtered through a 400 mesh (38 μm) screen because the maximum particle size that should be present is 40 μm. When required, the dry weight of this "coarse" material if significant, can be added to the

results obtained from the Coulter Counter if, for example, the % oil in a water layer is being determined.

Once the sample is properly screened it is sufficiently diluted in an aqueous electrolyte solution such that the particulates can pass through the sensing zone (aperture) one at a time. An electrolyte solution marketed by Coulter under the trade name "Isoton" is used, the composition of which is given below:

Sodium chloride.....7.93 g/l
Disodium EDTA.....0.38 g/l
Potassium chloride.....0.40 g/l
Monosodium phosphate.....0.19 g/l
Disodium phosphate.....1.95 g/l
Sodium fluoride.....0.50 g/l

The diluted sample is then added to more electrolyte such that the concentration of particulates is sufficient for analysis. This solution is then drawn through the aperture to obtain % volume and count data for each channel. Having these data plus the dilution factors and knowing the volume of sample analyzed (drawn through the aperture) allows calculation of % particulates in the original sample and the plotting of a size distribution graph. A computer program is used to simplify the calculations.

The size of oil particles present in the fluid sample to be analyzed should fall within the useful range of a single aperture tube. In cases where the size distribution is not within this range there may be a loss of fine particles below the lower limit or a loss of coarser particles above the higher limit. Therefore smaller, or larger aperture tubes, respectively, will be required.

TOTAL OIL ANALYSIS

The total oil analysis was performed in order to obtain an approximation of the percent oil or bitumen contained in the emulsion used for demulsifier testing. The procedures involved adding about 25 ml of emulsion sample to a clean and tared standard eight inch 400 mesh (38 μm) screen with a receiver pan. After determining the weight of the sample and allowing most of the sample to filter through the screen, the whole assembly was placed in an oven at about 90°C for about 16 hours. Further heating showed little additional weight loss. After cooling and weighing, the total

amount of bitumen can be calculated from the residue remaining both on the screen (>38 μm) and in the pan (<38 μm). The weight of the finer material should approximate that obtained by the Coulter Counter method.

This method is at best only an approximation of the % total oil in the emulsion since heating, if too excessive, can drive off the lighter, more volatile hydrocarbons in the sample while not enough heating will not remove all of the water.